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Salt Water Corrosion Characteristics of Specially Formulated Greases

Development of Grease Test Procedure and Test Results

Final Report

Prepared for:

U.S. Army Belvoir RD&E Center
Fort Belvoir, Virginia 22060-5606

Prepared by:

IIT Research Institute
10 West 35th Street
Chicago, Illinois 60616-3799

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Report No. IITRI-P06085-11

SALT WATER CORROSION CHARACTERISTICS OF SPECIALLY FORMULATED GREASES

Development of Grease Test Procedure and Test Results

Final Report covering the period
3 September 1987 to 2 September 1988

Prepared for:

U.S. Army Belvoir RD&E Center
Fort Belvoir, Virginia 22060-5606

Attention: Ms. JoAnn Noble
Materials, Fuels and Lubricants Laboratory

Prepared by:

B. Panda
IIT Research Institute
10 West 35th Street
Chicago, Illinois 60616-3799

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To develop a more reliable, effective, and sensitive method for testing the corrosion protective properties of greases, a dynamic test apparatus was designed and constructed. This development was carried out in two phases: During the first phase, some preliminary tests were performed to identify sensitive parameters that could be monitored for better evaluation. Based on these results, the test design was modified and a clear-cut experimental procedure was formulated for the second-phase tests.

Results obtained by the established test procedure, indicated by the average axial thrust drop with time measure on the loaded test bearing, showed good reproducibility and can characterize various greases used by the Army.

Small quantities of inhibitors like calcium and barium sulfonates and Lubrizol 5142 were added to three candidate greases. A small reduction in the corrosion-preventive properties (continued over)

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19. ABSTRACT (continued)

resulted when the sulfonates were used (both calcium and barium sulfonates yielded similar resultes), whereas Lubrizol really improved this property.

PREFACE

This final report, "Salt Water Corrosion Characteristics of Specially Formulated Greases" summarizes the efforts carried out by IIT Research Institute (IITRI) under Contract DAAK70-87-C-0047 toward the development of test setup and procedure for testing greases against salt water corrosion. In addition, the report presents results of tests performed on candidate greases and the ones formulated at IITRI. The entire task was carried out during the period 3 September 1987 to 3 September 1988. The internal designation of this report is IITRI-P06085-11.

The results indicate that a viable and direct method to characterize various greases against seawater corrosion was developed which is able to distinguish among greases by virtue of their corrosion resistance. A wide variety of greases has been tested in this program, indicating the effectiveness and viability of the developed test procedure.

We acknowledge with gratitude the support from the U.S. Army at Fort Belvoir and the critical evaluations and suggestions offered by Ms. JoAnn Noble, Mr. Schaekel, Mr. Maurice LePera, and Dr. Spitzer at various stages of this program. At IITRI, technical contributions were offered by Dr. Suresh K. Verma, Mr. Edward J. Vesely, Jr., Mr. James Cheng, and Mr. Tom Todner. Editing of this report was done by Ms. Violet Johnson and word processing by Ms. Gail Rardin and Ms. Cathy Machaj.

Respectfully submitted,

IIT RESEARCH INSTITUTE

Binayak Panda

Binayak Panda
Research Engineer

Approved:

Suresh K. Verma

Suresh K. Verma, Director
Surface Engineering Center

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1. INTRODUCTION

Concerns regarding ASTM D 1743, Standard Test Method for Corrosion Preventive Properties of Lubricating Greases, are many. In order to make this test method easy and less controversial, it is important that some of these concerns be removed and that the test procedure be reevaluated.

Among other things, there is the problem of test bearings. The test bearings used as standards have become obsolete. As a result, these bearings are beginning to form stain on their contact surfaces which is expected to affect the evaluation process seriously. The revised draft of the aforementioned test procedure includes another set of cone and roller assembly (LM 11949 and LM 11910), which counters the above problem but does not address some of the other concerns with this test procedure. Perhaps the most important concern is the static condition under which the corrosion product develops on the test bearing surface. The dynamic conditions under which the bearings function during service distributes the corrosion protective grease uniformly and also maintains a thinner film of grease under higher service loads.

Another concern with ASTM D 1743 test method is the problem of keeping the cup and cone of the grease filled bearing together during testing; momentary separation increases corrosion in areas where the rollers contact the cup. Corrosion marks on the cup become even more complicated by the presence of staining materials in the test greases. No suitable solvent has yet been found to remove such stains.

In view of the concerns mentioned above, the U.S. Army launched a program to develop a new method of grease testing altogether, which would possibly overcome most of these problems. This report describes the results of efforts made by IITRI to develop this new test procedure.

2. IITRI APPROACH

Eliminating the concerns described and arriving at a popular test method is not easy. However, a new test procedure introducing proper test environment, corrosion under stress and motion, and quantitative corrosion damage evaluation on test bearings can evolve into a well-accepted test procedure. The following paragraphs describe how the test procedure was designed to incorporate these developments.

2.1 SELECTION OF TEST BEARINGS

In line with the Army recommendation, roller assembly LM 11949 and the cup LM 119101 were selected as the test bearings. These bearings were inexpensive and were plentifully available.

2.2 EVALUATION OF CORROSION DEGRADATION

The corrosion process consumes the iron from the bearing steels by converting it to iron oxide. The corrosion process, therefore, could be conveniently and accurately monitored by the weight loss measurements provided that the products of corrosion are removed effectively. When the corrosion is not localized (pitting is a form of localized corrosion), change in dimensions could be used as a measure of corrosion. In addition to these measurements, electrochemical methods of corrosion current and potential measurement are often used. Electrochemical measurements provide the magnitude of instantaneous corrosion loss. Due to complexities of sample geometry, the dynamic nature of testing, and presence of a thin film of grease on the corroding surface, however, electrochemical measurements were not favored for this study. Although this method is expected to provide data on corrosion rates accurately within very short time, its feasibility needed extensive efforts. With the electrochemical studies ruled out, some preliminary experiments were defined to establish the best parameters for corrosion evaluation. Some of the selected parameters are:

- weight change
- dimensional change
- change in frictional losses
- change in the color of grease
- change in pH of the corroding environment.

2.3 SELECTION OF CORRODING ENVIRONMENT

Corrosion of grease-lubricated bearings in the marine atmosphere was the main concern to the Army. Besides, there was no well accepted method to test the grease against salt-water corrosion. Synthetic seawater was, therefore, chosen as the corroding medium. To accelerate the corrosion process as a means of reducing the testing time, undiluted seawater was selected as the sole corroding medium for the entire test matrix.

2.4 STRESS AND MOTION DURING CORROSION TESTING

Stress on the bearing surfaces plays a dominant role in the corrosion of bearings. It is therefore important to test the bearings coated with the test grease under stresses comparable to those expected in service.

Similar to the stress, the motion of the rollers on the test bearing surface causes significant changes in the corrosion rates and the nucleation and growth of corrosion pits. Theoretically, introduction of motion in dynamic corrosion testing reduces the probability for the pit growth. Pits found under these conditions are shallow and are uniformly distributed.

In line with Army requirements, the test procedure was designed to incorporate grease testing under dynamic motion and stress. The following sections describe the design of the test apparatus incorporating the above testing criteria.

3. DESIGN AND DEVELOPMENT OF TEST SETUP

The main thrust of this development has been the dynamic testing and the elimination of operator involvement in evaluating the extent of corrosion to the test bearings. These considerations called for the design of a test setup that can corrode the test bearings under stress and motion. The extent of corrosion on the test bearings could then be evaluated by one or more of the following methods:

(a) after a thorough cleaning, the weight loss of the outer race

(b) change in axial dimension of the test bearing by measuring the actual physical displacement or by measuring the change in axial thrust in a preloaded bearing with time

(c) increase in frictional losses due to progressive corrosion

(d) change in the color of grease on the test bearing due to the mixing of corrosion products with it

(e) change in pH of the corroding solution.

The development of the test setup can be divided into two phases. During Phase I the test setup was designed and fabricated; some trial runs were made, and some of the above corrosion parameters were measured to find out the most effective way to evaluate corrosion on test bearings. Experience gained from Phase I was used to modify the test setup and procedure for Phase II tests.

3.1 PHASE I TESTS

Figure 1 is a photograph and Figure 2 an assembly drawing of the test apparatus as first designed. This setup was subsequently modified for improved performance during Phase II. The apparatus consists of two rugged 2 in. thick plates supported by four columns of exactly equal lengths. The drive motor, which is coaxial to the test bearings, is mounted on top of the top plate. The drive system for the test bearings consists of three components for assembly convenience, the one closest to the test bearing being made out of stainless steel. Although 316 stainless steel was used for this construction, the material specification was later changed to 431 SS for better wear resistance. The test bearing cone is press-fitted to the end of

this bearing drive shaft. The test bearing cup is press-fitted into the bearing cup housing shown in Figure 2. Both the bearing cup housing and its holder contain the outer race and the corroding synthetic seawater, and the assembly can freely rotate about the thrust bearing at the bottom. To avoid spillage of the seawater, a Plexiglas cover (not shown in Figure 2) is screwed onto the top of the bearing cup housing.

The entire assembly can be moved up or down by rotating the two hex-nuts at the bottom of the load shaft. A load cell, placed between the bottom supporting plate and the hex-nuts, has the capacity to carry loads of up to 2000 lb. The thrust load exerted on the test bearing is measured by the load cell and can be monitored constantly.

During a typical test run, the grease-packed test bearing is thrust loaded and run in the presence of seawater leveled up to the top of the bearing cup housing. Although the applied grease tries to protect the bearing surfaces, corrosion sets in after a time and bring about changes in many measurable physical and chemical properties of the corroding system.

The results of the Phase I tests are reported in Section 5. During this phase, efforts were made to determine the most effective parameter for the corrosion evaluation. The following parameters were measured during this phase:

- change in the axial thrust with time
- change in the color of the grease
- change in the pH of the corroding seawater.

In addition, some efforts were made to study the changes in frictional losses with time.

3.2 PHASE II TESTS

The results of Phase I tests were in line with the theoretical expectations; however, for better accuracy and reproducibility, attempts were made to improve the rigidity and mechanical stability of the test setup. To achieve this, the machine was aligned with additional milling and grinding of some critical surfaces; the load shaft was rebuilt and its hole on the bottom plate was enlarged; the four supporting columns were reconstructed out of tubes having larger diameter. Removal of the thrust bearing from the bottom of the

bearing cup housing holder improved the mechanical stability measured by the run-out of the rotating shaft. All of these modifications resulted in 0.0004 in. run-out on the bearing drive shaft.

Phase I efforts pointed out the need for a more powerful motor for the apparatus; accordingly, a 1/2 hp motor was used for the drive train. Components exposed to salt water were made out of 316 SS; of these, the ones subjected to repeated wear needed to be wear resistant. The material for such components was changed to 431 SS which, after suitable heat-treatment, possessed adequate hardness and corrosion resistance.

Perhaps the most important experience gained from Phase I was the effect of the heat generated in the test bearings which was substantial and varied from grease to grease. The variation in temperature not only changed the film thickness of the lubricant and affected the corrosion kinetics, but also changed dimensions of components simply by raising their temperature. To counteract this effect, a cooling system was designed and placed on top of the bearing cup housing. This is illustrated in Figure 3, which also shows the appearance of the final modified test apparatus.

The cooling system consists of a see-through Plexiglas cylindrical outer cover containing the corroding seawater, bolted onto the bearing cup housing through a rubber gasket. The hot seawater, which is expected to convect upward toward the cooling system, transfers the heat to the copper annular cooling ring positioned to the rotating shaft inside the Plexiglas concentric. The cooling ring is cooled by the passage of cold tap water. The flow of tap water could be controlled depending on the heat generation so as to maintain the test apparatus at room temperature. To monitor the bearing temperature and to control the corrosion temperature, a fine Chromel-Alumel thermocouple was placed in contact with the outer race of the test bearing which could be monitored with time along with the thrust drop across the test bearings.

Phase II tests were carried out using the test setup described above and shown in Figure 3. The results of Phase II tests are given in Section 5.

3.3 OUTLINE OF TEST PROCEDURE

The following test procedure was used to obtain results during Phase II experiments.

3.3.1 Preparation of Bearing, Corrosion Cell, Cooling System, and Test Sample for Run-in

Degreasing the Test Bearing:

(1) Unwrap two bearings (2 cones and 2 races) and check the bearings for corrosion. If there is any evidence of corrosion, discard the bearing.

(2) Place the races and cones in a beaker containing 300 mL of acetone and sonicate for 30 min. After this, pour off the 300 mL of acetone. Add 300 mL of fresh acetone and sonicate for an additional 30 min. Pour off the 300 mL of acetone. Add 300 mL of alcohol to the beaker and sonicate for 30 min.

(3) After the alcohol cleaning, use forceps to remove the races and cones from the cleaning solution and place the parts on filter paper to air dry.

(4) Using forceps, weigh and record (to the nearest mg) the individual weights of each cone and race.

Cleaning the Corrosion Cell and Cooling System:

(1) All the metallic parts of the corrosion cell should be cleaned using acetone and isopropyl alcohol as the cleaning solutions. The cooling system should be cleaned with a suitable water-soluble liquid cleaner (e.g., Aquasol cleaner for ultrasonic cleaning).

(2) When required, use a fine mesh nylon pad or brush to remove grease and tenacious oxide or rust from the test cell and the cooling system.

(3) After washing each part in the sonicator, rinse the parts thoroughly under running tap water.

(4) Discard the detergent solution and fill the sonicator with distilled water. Sonicate the parts in distilled water for 30 min.

(5) Discard the distilled water and place the parts in beakers containing 300 mL of acetone. Sonicate for 30 min.

(6) After 30 min, pour off the acetone and add 300 mL of acetone to the beaker. Sonicate for 30 min and pour off the acetone.

(7) Add 300 mL of isopropyl alcohol to the beakers containing each part of the corrosion cell and the beaker containing the cooling system. Sonicate for 30 min.

(8) Wearing gloves, remove each component and air-blow to remove the alcohol rinse.

(9) Place each component in a desiccator until used (not more than 24 h).

Preparation of Test Sample (determine density of test grease):

(1) Weigh a test sample holder of known volume. Record the weight to the nearest mg.

(2) Fill test sample holder with test grease, taking care to eliminate air pockets, and level the surface of the grease with a spatula.

(3) Weigh the filled container, and record the weight to the nearest mg.

(4) Assume the volume of the grease in the roller element is equal to 3 cc.

(5) Calculate the density of the test grease (DTG) = (weight of grease + container) - (weight of container)/volume of test sample holder.

(6) The amount of test grease to be packed in the roller element is equal to GTG (grams of test grease). GTG = DTG x 3.00 cc.

3.3.2 Corrosion Test Procedure

To minimize the effects of temperature variations, the machine must be kept in a temperature-controlled room. Since the motor generates heat during operation, it should be running at half its maximum speed for at least 12 h prior to the resumption of any testing. The following test procedure was followed during the Phase II experiments:

(1) Slide in the cooling system onto the bearing drive shaft, and then press-fit the test bearing cone onto it.

(2) Apply the entire amount of weighed test grease between the rollers of the cone.

(3) Press-fit the test bearing cup into the bearing cup housing, and fasten it onto its holder using the four screws. Although the joint between these two components is watertight, applying a thin layer of test grease between the contacting surfaces is recommended.

(4) Place the bearing cup assembly on the load shaft, and place the rubber gasket on the assembly with its holes properly aligned and a thin layer of test grease applied to both sides.

(5) Fill the bearing cup housing up to a level of about 1 cm from the top with synthetic seawater. Place the cone assembly onto the cup with care and without any trapped air bubbles under the cup and cone assembly.

(6) The cooling system can now be fastened to the top of the bearing cup housing, and the water hose and the thermocouple can be connected.

(7) Unlock the hex-nut, and raise the entire assembly by turning the hex-nut until the bearing drive shaft is firmly in contact with the motor drive shaft through the guide pins. If the motor is running, it should be turned off during this step.

(8) The motor should now be turned on and the speed regulator turned clockwise until 50% of the motor speed (rpm) is reached.

(9) Load the test bearing up to about 400 lb, and note the start of run-in time. The cooling system should now be filled with synthetic seawater through the annular space between the drive shaft and the Plexiglas cooling ring.

(10) Run the system through the run-in time of 1 h by adjusting the volume of cooling water such that the temperature of the testing cone remains at room temperature and by gradually raising the thrust load to 500 lb within the first 30 min of run-in.

(11) At the end of the run-in period, the water level and the thrust load should be adjusted by adding seawater to the previous level and by tightening and locking the nut at 500 lb thrust load.

(12) The test should run for 48 h beyond the run-in time, and the thrust load and bearing temperature should be monitored constantly during this time. Any loss of seawater (except for leaks) will be assumed to be due to evaporation and will be replenished by adding distilled water. At the end of the test, the machine should be stopped, the cell removed, and its components cleaned.

(13) The corrosion protection ability of a grease sample will then be given by the average thrust load loss expressed in units of lb/h over the entire test period. To calculate the results of the mass loss of the bearing cup, the part should be thoroughly cleaned with a nylon brush or cloth. The difference in weight gives the mass loss over the entire period for both the corrosion and wear.

4. PROCUREMENT AND FORMULATION OF TEST GREASES

Three greases with QPL Nos. M-7701, M-7703, and M-7707 were chosen as the three base-line greases. These greases were procured from their manufacturers: Battenfeld Grease and Oil Corporation of New York and Witco Chemical Corporation. To study the effects of corrosion inhibitors, a special grease was formulated and supplied to us by Battenfeld wherein the corrosion inhibitor was not added to an otherwise M-7707 grade high temperature grease. These four greases were the test materials for the entire program.

In addition to the above, more grease samples were supplied by the Army, and these were tested in the IITRI-developed test apparatus--Mobil WTR grease and Chevron SRI grease being two of them.

To improve the salt water corrosion resistance of the above greases, the following inhibitors were selected and were added to the extent indicated:

Inhibitor	% Added
Neutral calcium sulfonate	1.0
Neutral barium sulfonate	1.0
Lubrizol 5142	2.0

These inhibitors were added to a small quantity of the base-line greases (50 g), and the compositions were mechanically mixed for two hours without application of any heat. A total of 13 different compositions were prepared for testing in this program, exclusive of the above-mentioned Mobil, Chevron, and other greases supplied by the Army.

5. RESULTS AND DISCUSSION

5.1 PHASE I

As mentioned earlier, the Phase I experiments were oriented toward identifying parameters important in evaluating the corrosion-protective properties of candidate greases. The following parameters were selected for study as a function of time since the corrosion progressed in seawater, resulting in:

- change in thrust load
- change in pH
- change in grease color
- change in frictional losses.

The test procedure for this phase was different from the one described earlier in Section 3.3 for Phase II in the following manner:

(a) The thrust load was adjusted to only 400 lb since the heating effects raised the load levels to higher values.

(b) Heat generated in the test bearing evaporated a substantial amount of water; thus the seawater level went down as the testing progressed. This loss was not replenished because the pH measurements were being carried out simultaneously.

(c) The tests were interrupted to measure the pH and observe the color of the grease. After this was done, the bearings were assembled and loaded to the levels they had reached prior to the pH readings.

Four greases were evaluated by the above procedure: M-7701, M-7703, M-7707, and a grease having the same base composition as M-7707 but without the inhibitor package in it. For convenience, this grease has been designated "M-7707-NI." Figures 4 through 11 show the thrust loss and changes in pH with time.

For grease M-7701, Figure 4 shows the reduction in the thrust load and the pH with time. Although both drop with time, the thrust drop is more and, therefore, is expected to be more sensitive to corrosion effects. Figure 5 shows an even faster drop of thrust under similar conditions. After about 8 h running, the load was raised to 400 lb to see if such a large drop was an

instrumental error. The load started falling again from 400 lb at a rate initially similar to, and later faster than, the rate before the load increase.

Figures 6 and 7 show similar thrust and pH changes for grease M-7703. Both thrust and pH were evidently dropping with time when this grease was used. While the pH drop is perhaps as much as was observed with M-7701 grease, the thrust drop is not as great. Figure 7 shows results with 2.5 g of grease, and Figure 6 shows results with 2.0 g of grease. Generally, the thrust drop behavior in both cases is the same except for the drop observed during the first 5 h of testing. This difference may be attributed to thermal effects and will be discussed in greater detail later.

Grease M-7707-NI showed a smooth drop in thrust load and pH (Figure 10) but is ranked as inferior with respect to corrosion protection when compared with M-7707. To prove this point even further, two more tests were carried out under identical conditions and the results are plotted in Figure 11, which shows the scatter expected in the Phase I test procedure.

In addition to the pH and thrust drop, change in grease color was also monitored during this phase. Figures 12 to 30 are photographs of the color change in candidate greases under dynamic corrosion conditions. Color changes were due to the intermixing of the grease with the corrosion product, iron oxide. The color changes to brown when Fe_2O_3 with its associated water molecules forms as a corrosion product. This is expected when there is an abundance of oxygen available for corrosion. Lack of oxygen creates the black iron oxide, Fe_3O_4. Since corrosion inhibitors are often oxidizers, a black color of grease would indicate the lack of a corrosion inhibitor.

Figures 13 to 17 are photographs of the grease color changes during the first 9 h of testing in seawater for M-7701 grease. As is evident from these photographs, the brown rust forms within the first 2 h of testing and the black corrosion products form sometime between 2 and 4 h. After 4 h of testing, the color turns increasingly black.

Figures 18 to 23 show the progressive color change in the test grease M-7703 due to the intermixing of iron oxide. A very small quantity of iron oxide develops in this grease within 2 h of testing, and the amount of brown color development is comparable to that of M-7701 after only 4 h of testing.

With the corrosion rate being slower than for M-7701, the black color develops sometime between 7 and 10 h of testing.

Figures 24 to 27 show the color change in the high-temperature grease M-7707. In this case, both the brown and the black colors develop sometime between 4 and 7 h of corrosion testing. When the corrosion inhibitors are removed from this quality grease, extensive brown rust is formed within 2 h of testing and the black color develops sometime between 2 and 4 h.

Results on the M-7707-NI grease are presented in Figures 28 to 30.

From these studies involving the color change, the following characterizations can be made:

- Both M-7701 and M-7707-NI grease have nearly the same corrosion protection capability, which is clearly inferior to that of M-7703 and M-7707 greases.
- Both M-7703 and M-7707 possess superior corrosion protection properties. M-7703 allows corrosion at a very slow rate but starts corrosion early. On the other hand, M-7707 protects the bearing for a longer period but once corrosion starts, it progresses fast.

During Phase I, a few weight loss (due to corrosion) measurements of the test bearing cup were carried out with encouraging results and were, therefore, incorporated throughout the Phase II experiments. In addition to all these, some frictional loss measurements were carried out by measuring the voltage drop across the d-c drive motor. The results were inconclusive and are not reported here.

5.2 PHASE II RESULTS

Phase I results clearly show that the corrosion protective properties of lubricating greases can be evaluated by conducting thrust loss experiments as described. All three parameters--thrust loss, weight loss, and color change--could be used for corrosion evaluation of the test bearings and, hence, protective properties of the test greases against salt water. Based on these observations, Phase II experiments were carried out using thrust and weight loss as corrosion evaluation criteria, since the judgment on color change may lead to controversy. To eliminate the effects of ambient and transient temperature effects, the whole setup was kept in a temperature-controlled atmosphere with a cooling system incorporated. Also, the rigidity of the system was improved by changing some of the parts and the four support pillars. Phase II, therefore, began with tests carried out at room temperature. Since higher thrust loads could be tolerated under these conditions, initial thrust load was raised to 500 lb. Also, since the corrosion kinetics at lower temperature were expected to be low, the test duration was 48 h.

Table 1 shows data obtained on the various procured greases and Figures 31 to 35 show thrust drop behavior of these greases. Some of these tests were

TABLE 1. THRUST LOAD LOSS AND WEIGHT LOSS DATA FOR CANDIDATE GREASES

Grease	Test No.	Weight Loss,[a] g	Thrust Loss, lb	Test Time, h	Avg Thrust Loss Rate, lb/h	Avg Mass Loss Rate, g/h x 10^4
Mobil WTR Grease	1	-	395	44	8.97	-
	2	0.0344	393	37	10.62	9.3
M-7707-NI (without inhibitor)	1	0.0300	353	47	7.51	6.4
	2	0.0278	369	48	7.69	5.8
M-7703 (Witco Grease)	1	-	91	48	1.89	-
M-7701	1	0.0374	135	48	2.81	7.8
	2	0.0293	145	48	3.02	6.1
Chevron SRI grease 2	1	0.0177	43	48	0.89	3.6
	2	0.0222	71	48	1.48	4.6

[a]Weight loss = (initial weight of cup) - (final weight of cup).

repeated to demonstrate the reproducibility. The thrust drop data in Table 1 indicate that the best protection to the bearings is offered by the Chevron SRI grease 2 followed by the Witco grease (QPL M-7703). The poorest performance was observed with Mobil WTR grease, followed by the slightly better high-temperature grease without inhibitor (M-7703-NI). The weight loss data do show a maximum value for the WTR grease and a minimum value for the Chevron grease, but there is large scatter. The main reasons for this scatter may be:

- The weight of the cup is large and change in weight is very small by comparison.
- Wear loss during the run-in period (where the loss is expected to be maximum on an hourly basis) is also included in this weight loss data, whereas the thrust loss, which is monitored only after the run-in period, does not reflect it.

The effects of the three corrosion inhibitors selected for this program were then evaluated, and the results are reported in Table 2. Figure 36 presents the thrust loss data for M-7701. For this grease, calcium and barium sulfonates are similar in effect but seem to reduce the corrosion protection (although the weight loss data do not indicate this). Lubrizol 5142 (2%) showed improved corrosion protection.

TABLE 2. EFFECT OF INHIBITORS ON GREASE M-7701

Inhibitor	Test No.	Weight Loss,[a] g	Thrust Loss, lb	Test Time, h	Avg Thrust Loss Rate, lb/h	Avg Mass Loss Rate, g/h x 10^4
None	1	0.0374	135	48	2.81	7.8
	2	0.0293	145	48	3.02	6.1
1% Ba sulfonate	1	0.0469	208	46	4.52	10.2
1% Ca sulfonate	1	0.0301	211	45	4.68	6.7
2% Lubrizol 5142	1	0.0192	113	48	2.35	4.0

[a]Weight loss = (initial weight of cup) - (final weight of cup).

The effect of inhibitors was also evaluated on the high-temperature grease M-7707. The required amount of inhibitors was added to the regular grease conforming to MIL spec. The test results are presented in Table 3 and Figure 37. Here again, Lubrizol 5142 showed the best results. The position of these curves with respect to the performance of the original grease cannot be evaluated since no data were generated for the M-7707 grease. Comparing the results of M-7707-NI grease containing no inhibitor at all, it is seen that all three of these formulations with inhibitor have superior corrosion resistance.

TABLE 3. THRUST LOAD LOSS AND WEIGHT LOSS DATA FOR GREASE M-7707 WITH INHIBITORS

Inhibitor	Mass Loss, g	Thrust Loss, lb	Test Time, h	Avg Thrust Loss Rate, lb/h	Avg Mass Loss Rate, g/h x 10^4
1% Ba sulfonate	0.032	73	48	1.52	6.6
1% Ca sulfonate	0.108	120	48	2.5	22.5
2% Lubrizol 5142	0.062	55	48	1.14	12.9

One test run per inhibitor.

Calcium and barium sulfonates were added to M-7703, and the test results are shown in Figure 38 and Table 4. Addition of these inhibitors decreased the corrosion-protective properties of M-7703. As mentioned earlier, similar results were observed with M-7701 grease.

TABLE 4. THRUST LOAD LOSS AND WEIGHT LOSS DATA FOR GREASE M-7703 WITH INHIBITORS

Inhibitor	Mass Loss, g	Thrust Loss, lb	Test Time, h	Avg Thrust Loss Rate, lb/h	Avg Mass Loss Rate, g/h x 10^4
None	-	91	48	1.89	-
1% Ba sulfonate	0.0507	162	48	3.37	10.5
1% Ca sulfonate	0.0606	145	48	3.02	12.6

One test run per inhibitor.

6. CONCLUSIONS

Efforts in this program generated a viable test technique able to distinguish between the corrosion protective properties of lubricating greases. A few developmental stress tests were employed to improve this test procedure, which yields better resolution than the existing ASTM D173 technique and incorporates direct testing of the grease in corrosive media.

The method characterizes the grease by virtue of the drop in axial thrust of a loaded test bearing in the presence of the grease; the faster the thrust load drop, the less is the corrosion protection. The procedure showed good reproducibility in duplicate tests.

Testing by this method shows that the corrosion resistance (conferred by the existing MIL spec greases could be improved by small additions of inhibitors. The most effective inhibitor was found to be Lubrizol 5142. Both barium and calcium sulfonates decreased the corrosion protection properties of the greases studied.

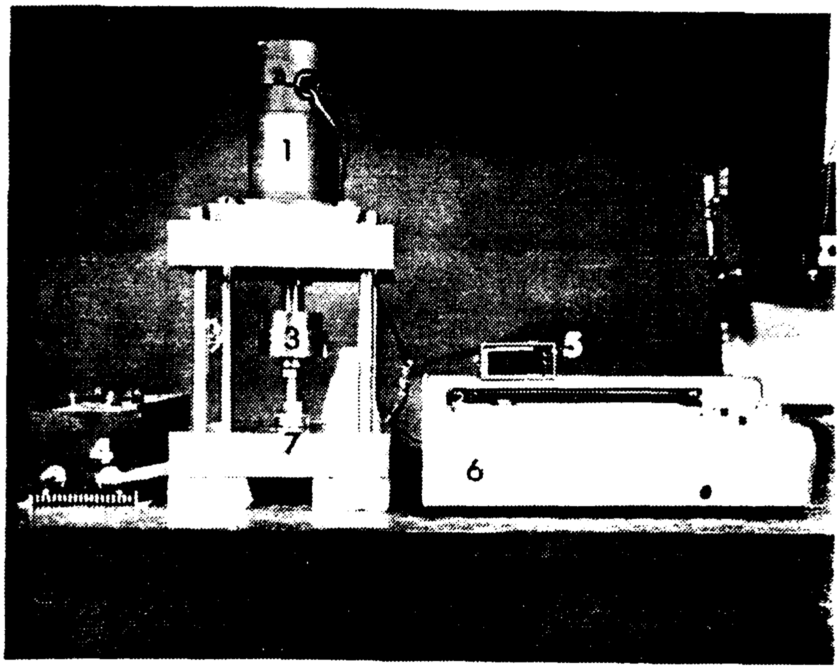
7. FUTURE EFFORTS

Corrosion in bearings is aggravated by the simultaneous action of wear. In lubricated bearings these two processes are affected by the changes in lubricant film and its stability. The overall corrosion is totally governed by the interplay of these three factors--corrosion, wear, and lubricant. The effects of these factors are difficult to separate and need extensive experimentation. It is therefore pertinent to study the effects of interplay of these parameters. While such experiments will aid in better understanding of the process, test procedures should be further developed to incorporate two different test temperatures because greases, after performing well at low temperatures, may break down at higher temperatures.

The program funded thus far has developed a new test machine that compares corrosion-protection properties of greases based on the thrust drop with time. Further work is required, however, to completely characterize the observed thrust drop for an in-depth understanding of this type of evaluation and for quantitative assessment of the contribution of the three interacting factors (corrosion, wear, and lubricant).

Neither the ASTM (static corrosion test) nor the IITRI-developed dynamic test provides the ideal simulation of the bearing corrosion occurring in Army vehicles, which experience both static and dynamic corrosion in service. This combination of static and dynamic corrosion could easily be achieved by modifying the IITRI-developed test apparatus. Such experiments could also correlate the static and dynamic tests. Since the modes of bearing failure are different for the static and the dynamic tests, it is necessary to conduct both the static and dynamic tests in combination with the static-dynamic modes for better simulation.

Statistical analysis of the results obtained by the developed test procedure is an important aspect to establish the efficacy of a test procedure. The establishment of variance and confidence levels is another aspect for future efforts.



1 Motor
2 Supporting Columns
3 Sample and Sample Support Cup
4 Power Controller
5 Load Indicator
6 Two Channel Recorder
7 Load Cell

Figure 1. Photograph of the first test [illegible]

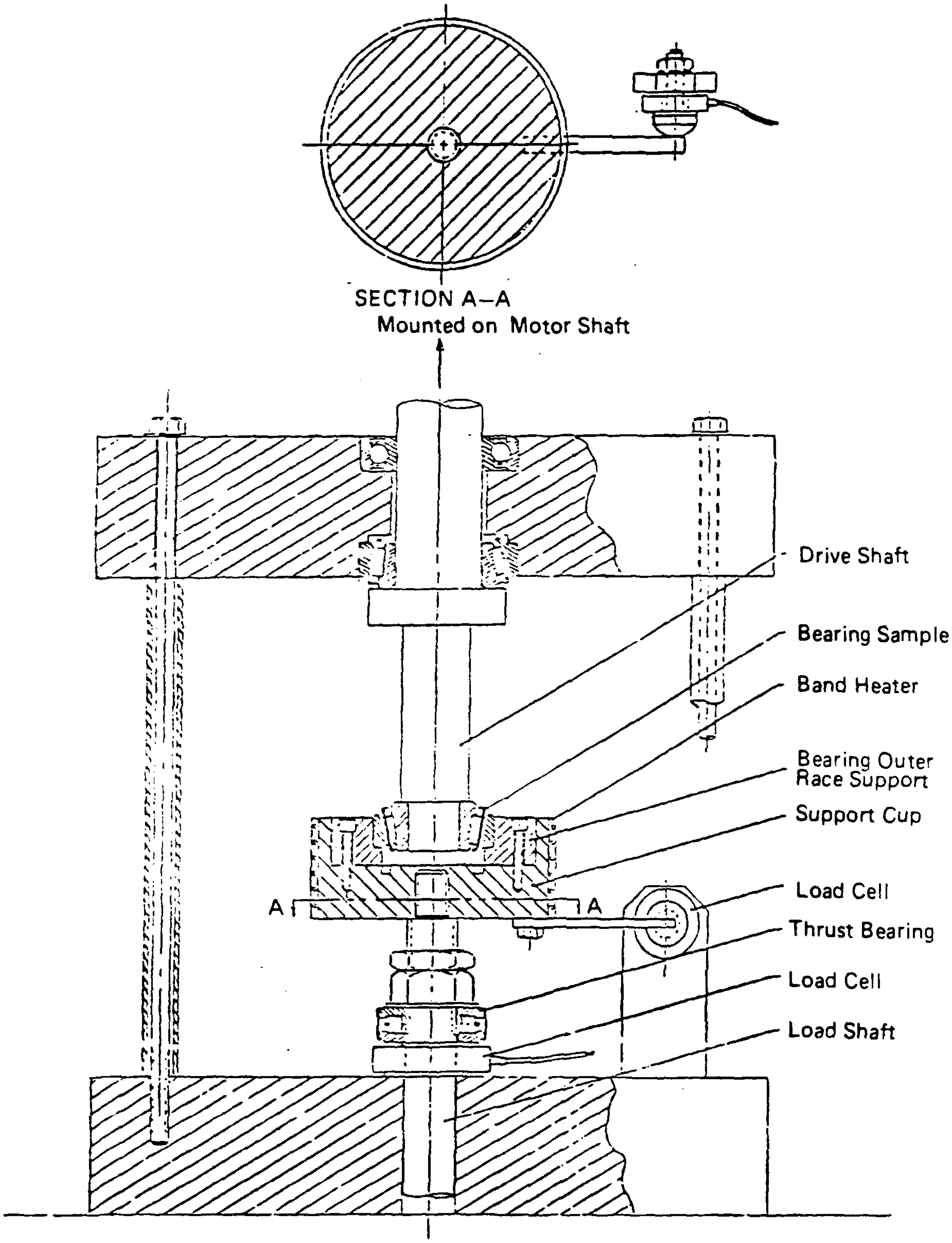
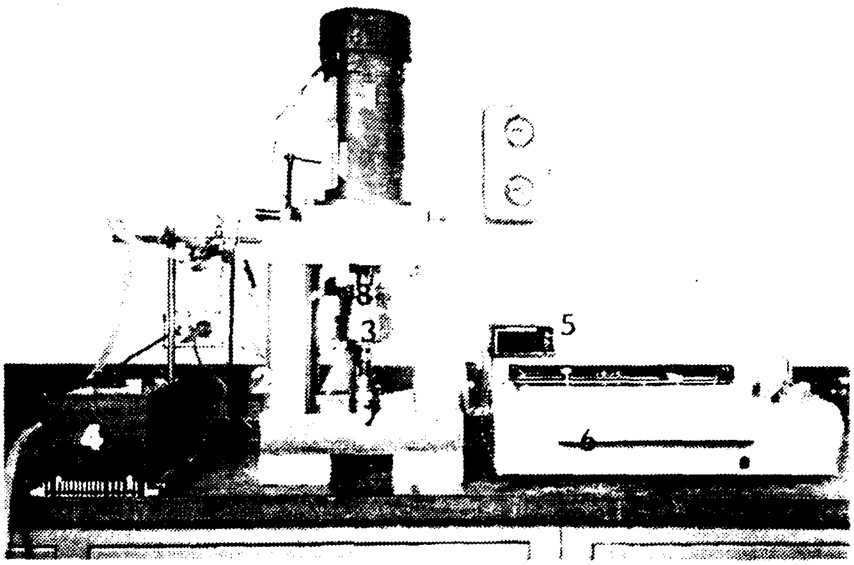


Figure 2. Assembly drawing of the first test apparatus designed.



1 Motor
2 Supporting Column
3 Sample and Sample Support Cup
4 Power Controller
5 Load Indicator
6 Recorder
7 Load Cell
8 Cooling System with Water Tubing

Figure 3. Photograph of the modified test apparatus used for Phase II experiments.

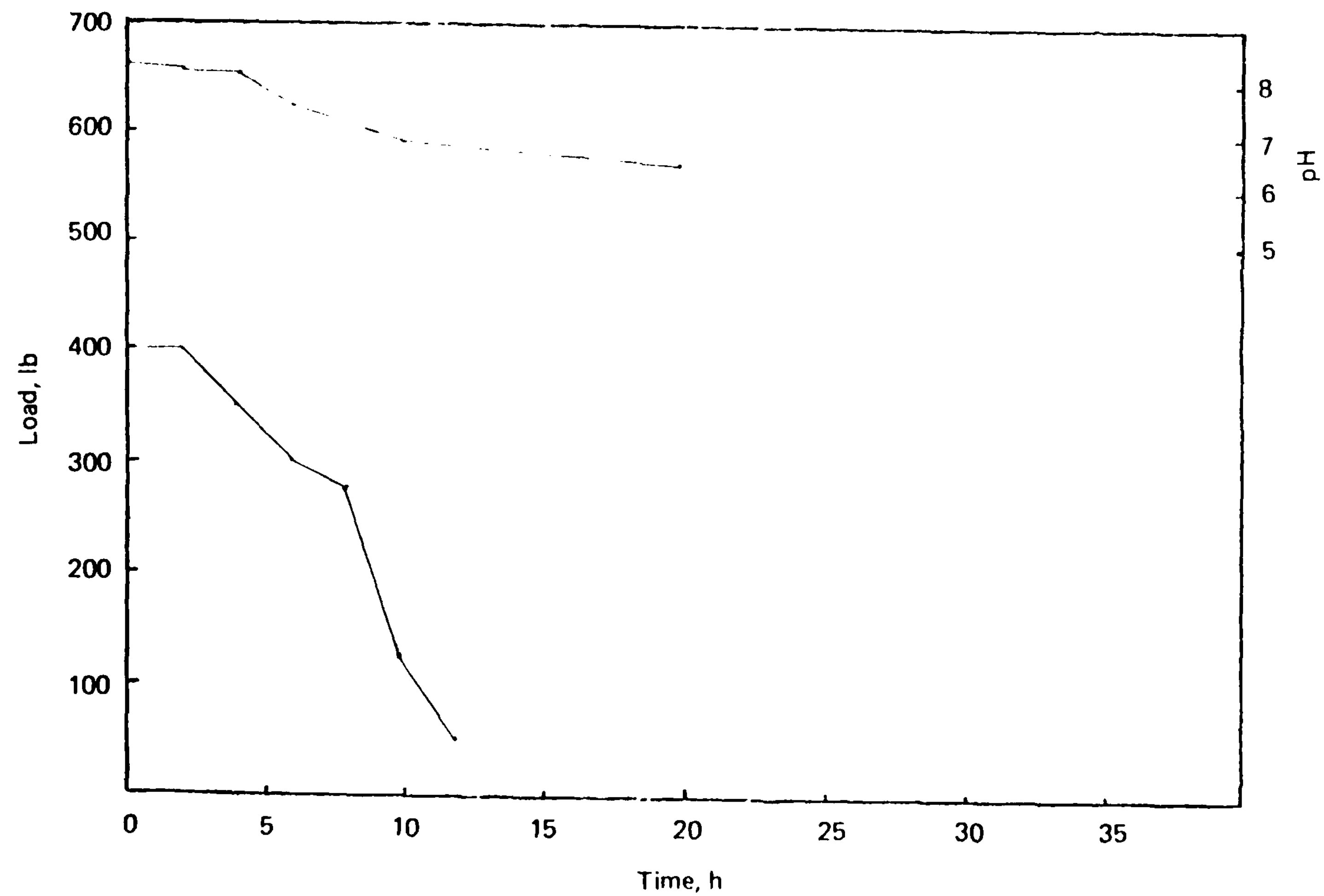


Figure 4. Change in axial thrust and pH with time for grease M-7701.

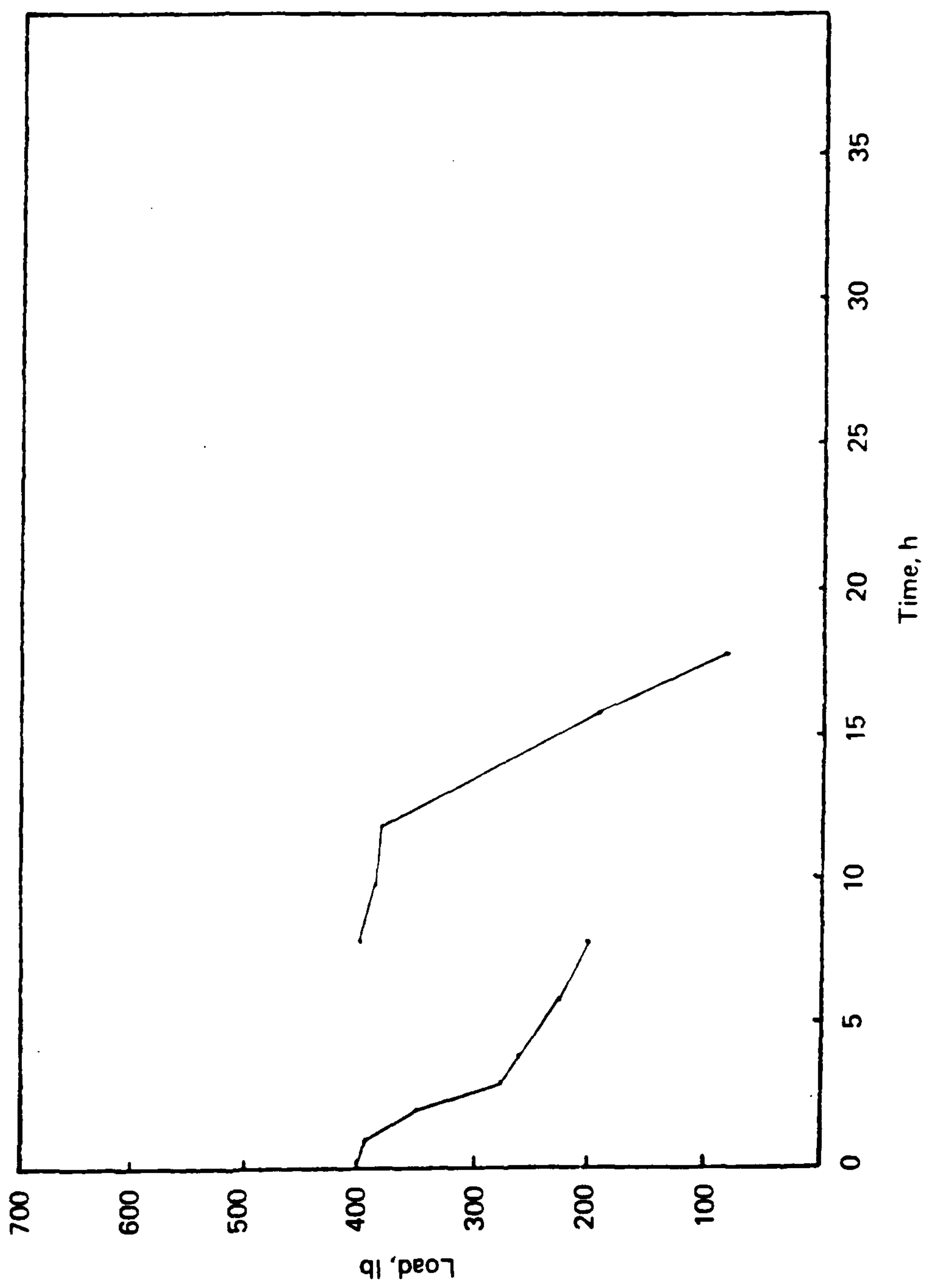


Figure 5. Change in axial thrust with time for grease M-7701 with a reloading after 8 h of testing.

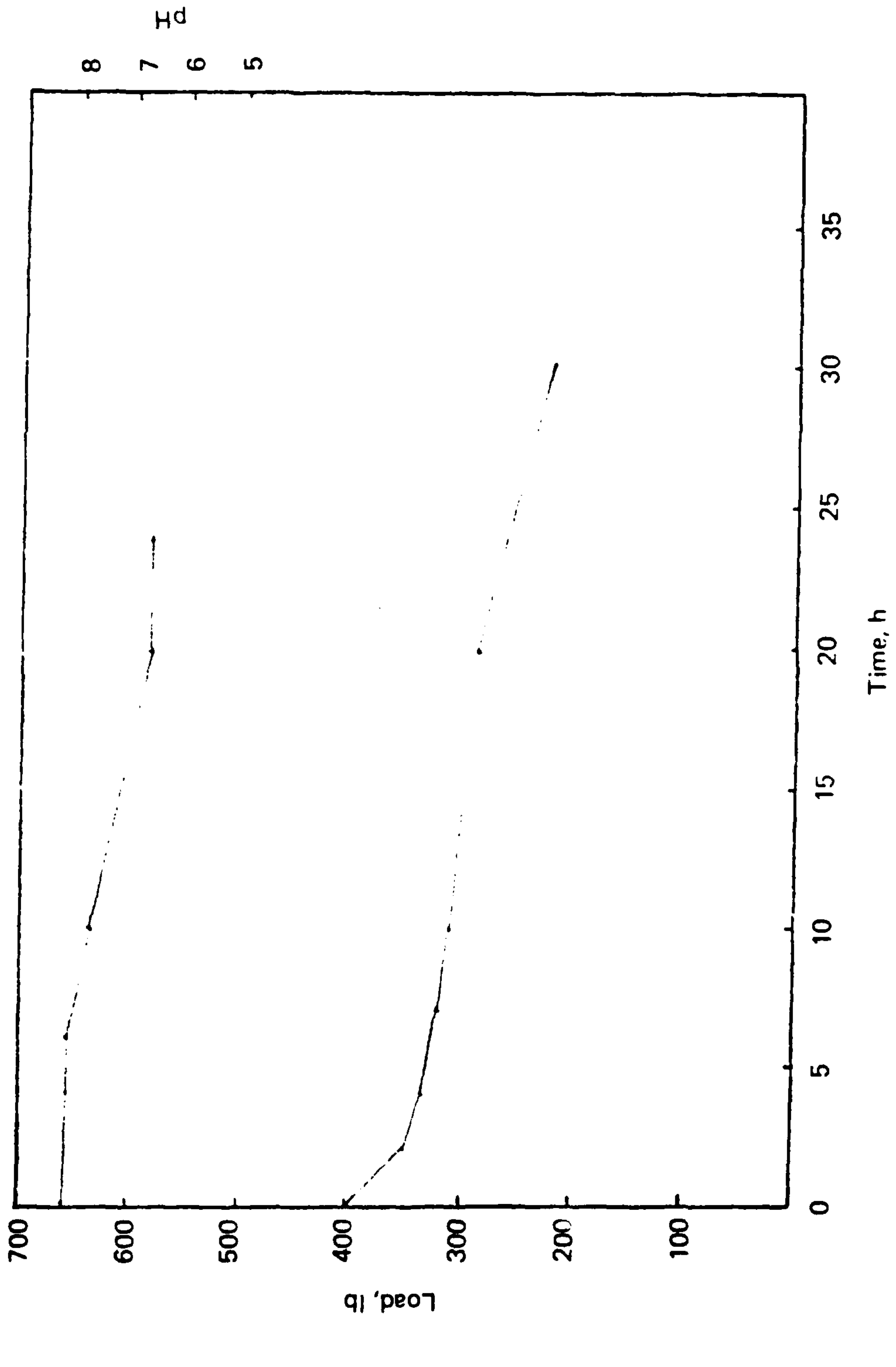


Figure 6. Change in axial thrust and pH with time for grease M-7703 with 2 g of test grease.

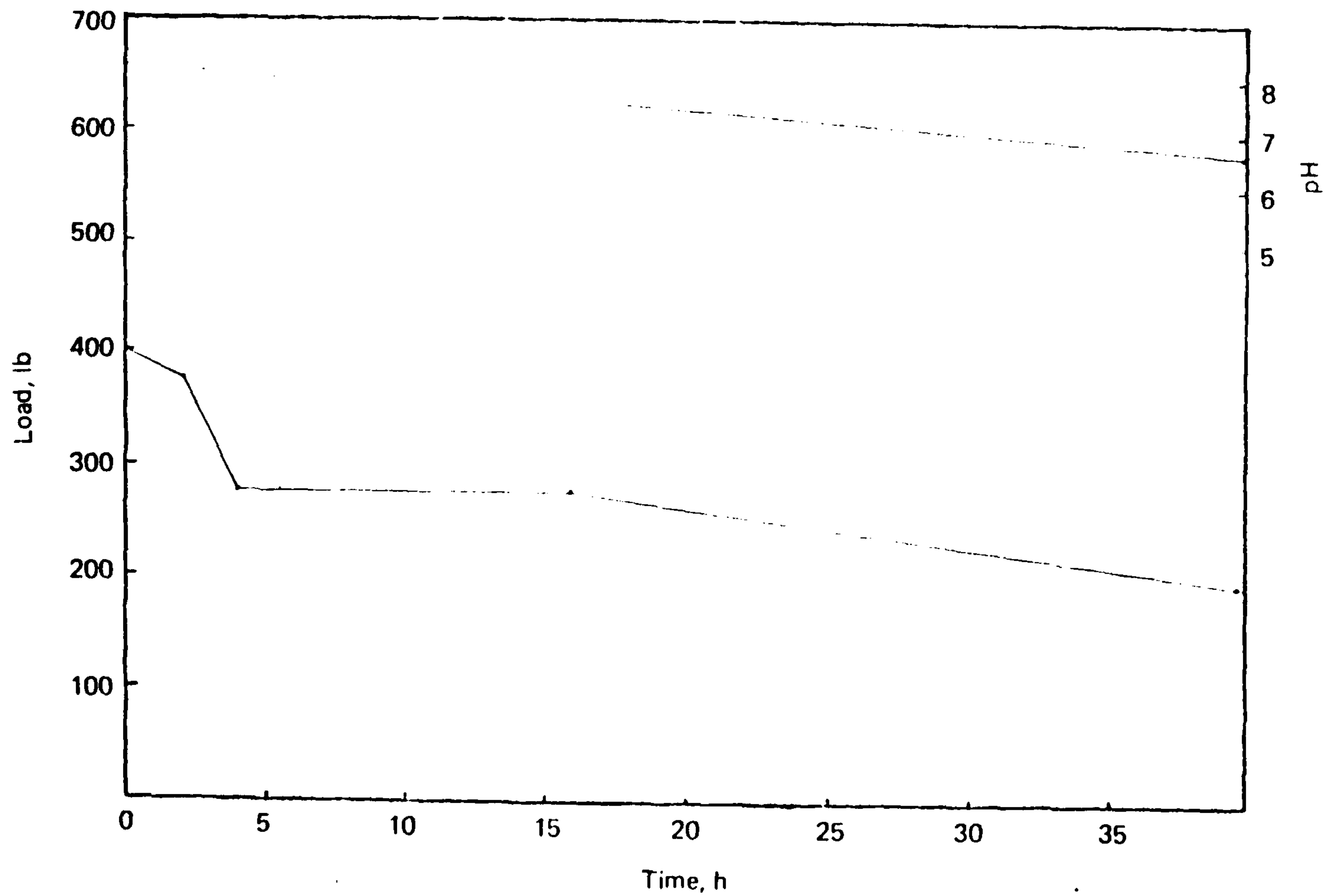


Figure 7. Change in axial thrust and pH with time for grease M-7703 (same conditions as in Figure 6) but with 2.5 g of test grease.

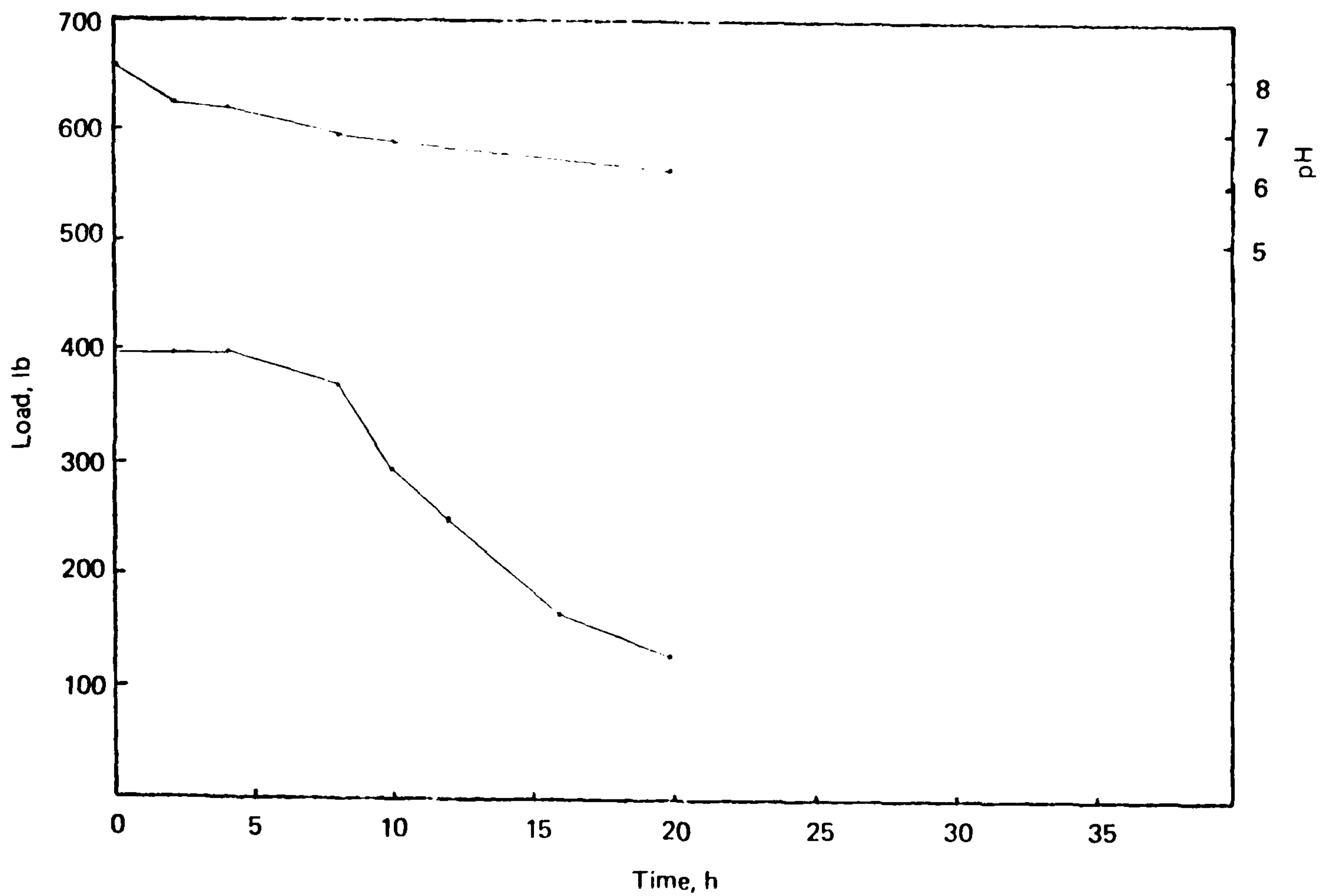


Figure 8. Change in axial thrust and pH with time for grease M-7707 with 2 g of test grease.

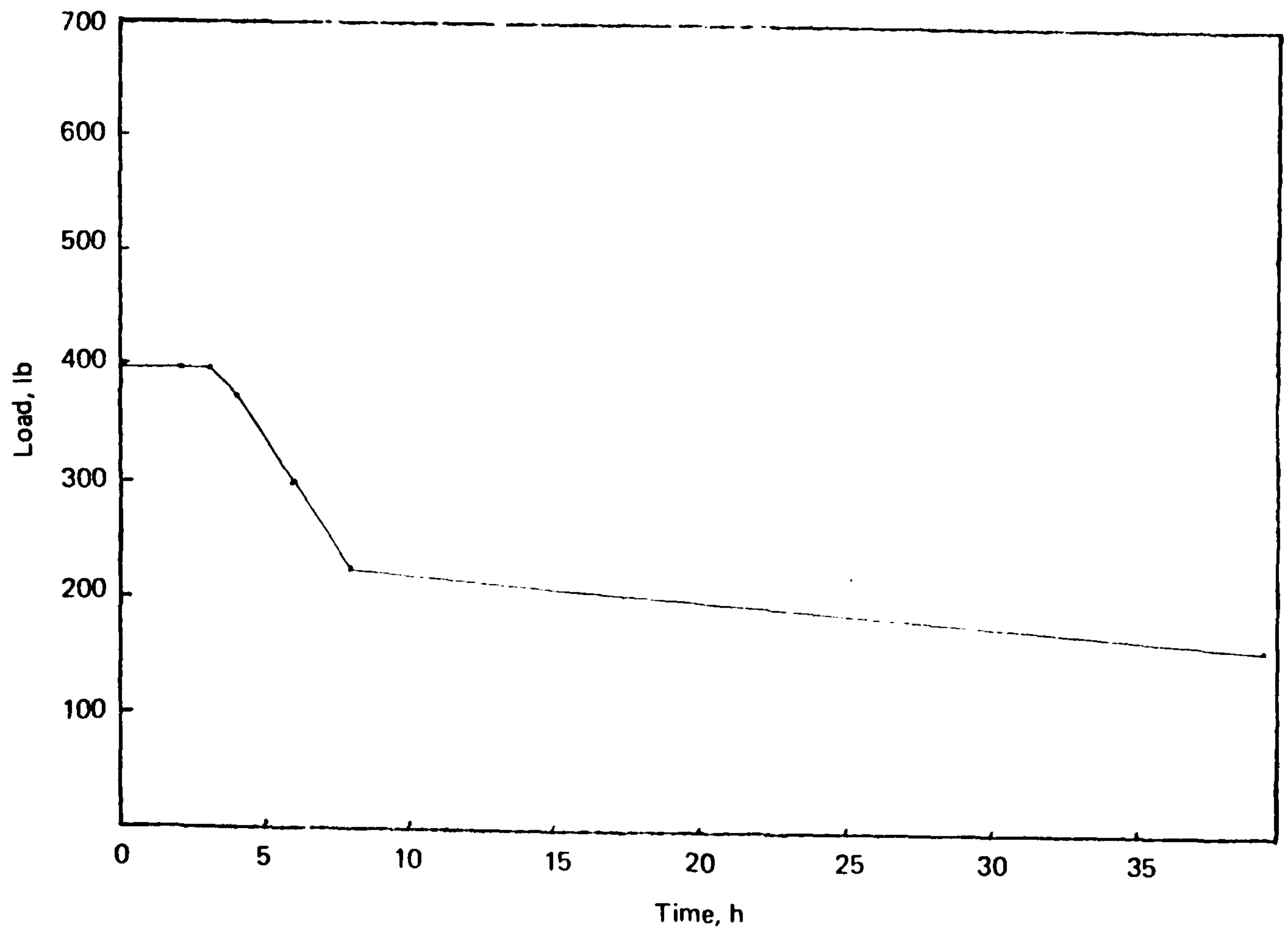


Figure 9. Change in axial thrust with time for grease M-7707 (same conditions as in Figure 8) but with 2.5 g of test grease.

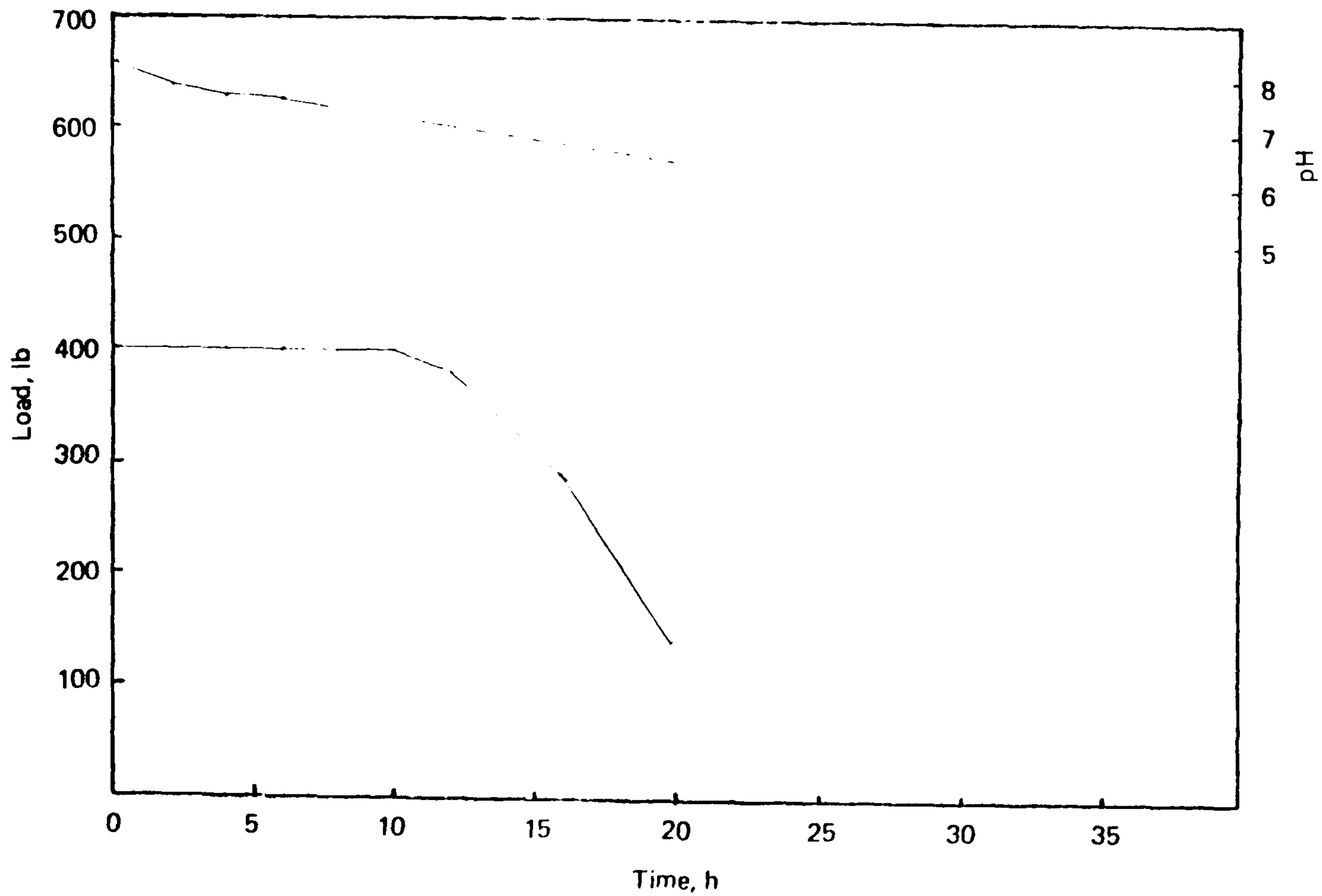


Figure 10. Change in axial thrust and pH for specially formulated grease M-7707-NI (M-7707 composition but without inhibitor).

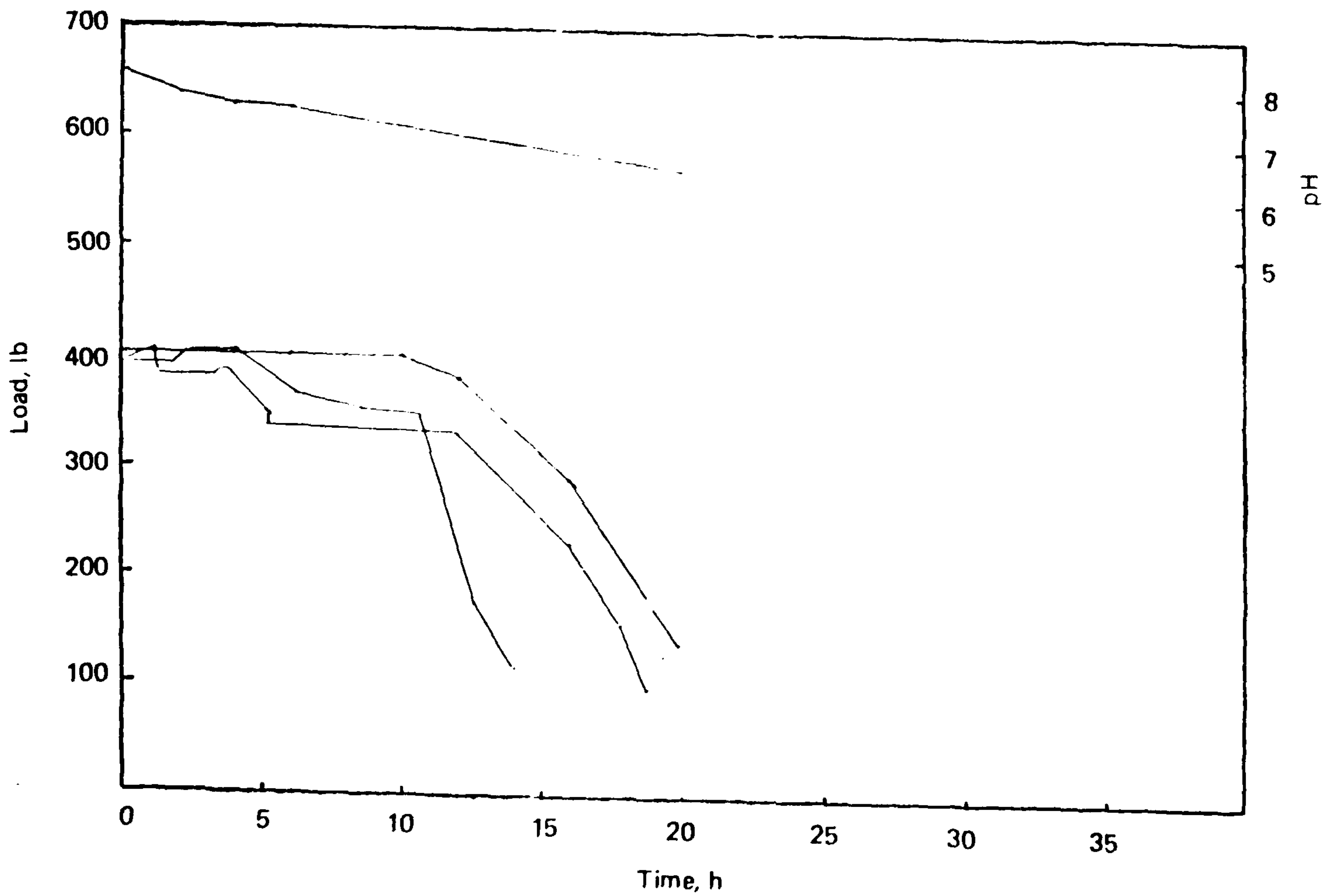
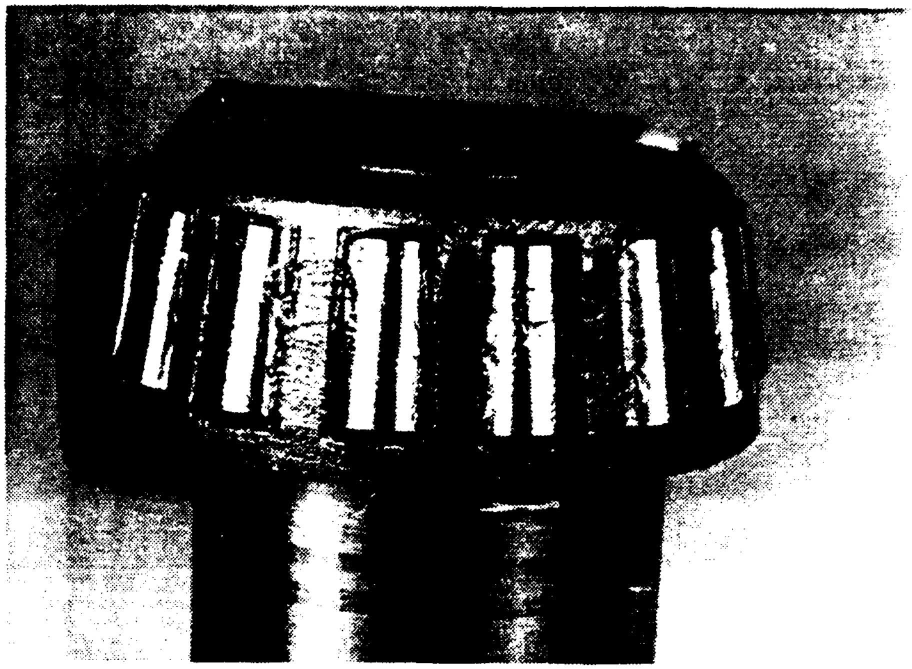


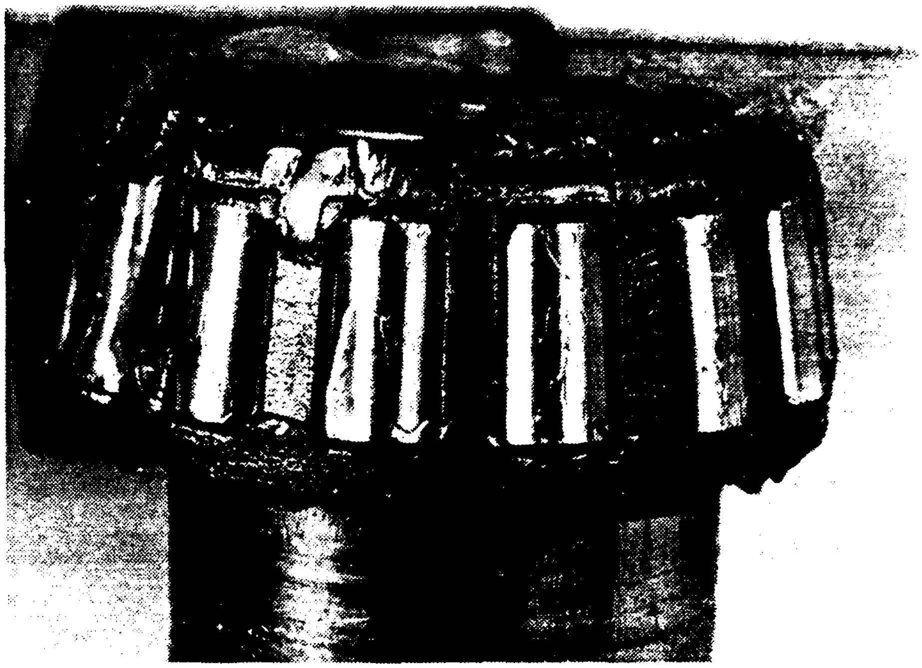
Figure 11. Change in axial thrust and pH for grease M-7707-NI (same conditions as in Figure 10), showing three axial thrust curves to indicate reproducibility.



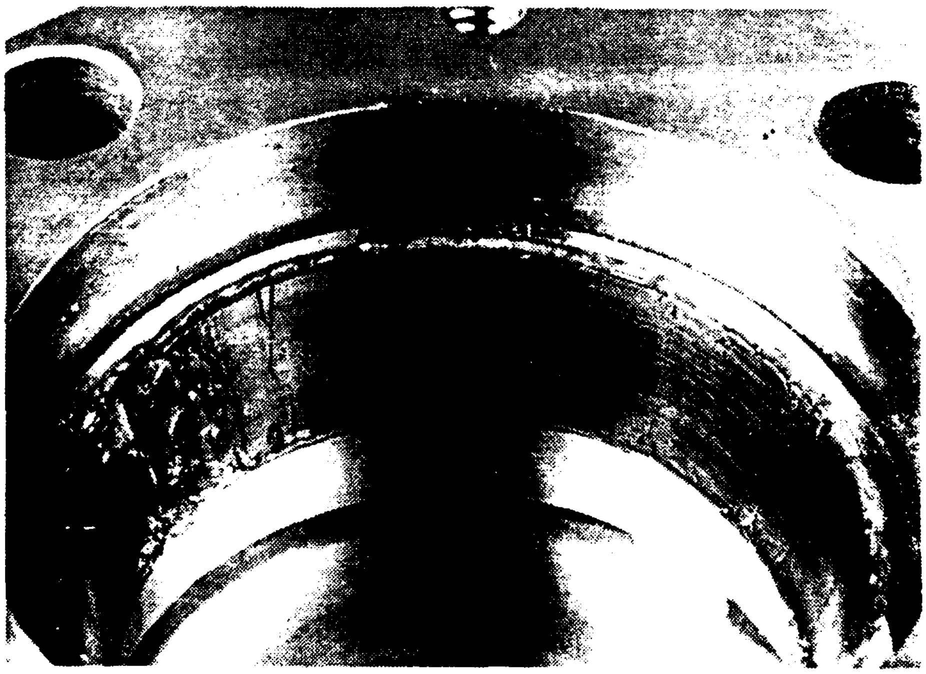
(a)

(b)

Figure 12. Appearance of test bearings ([illegible] cone) prior to testing grease M-[illegible].

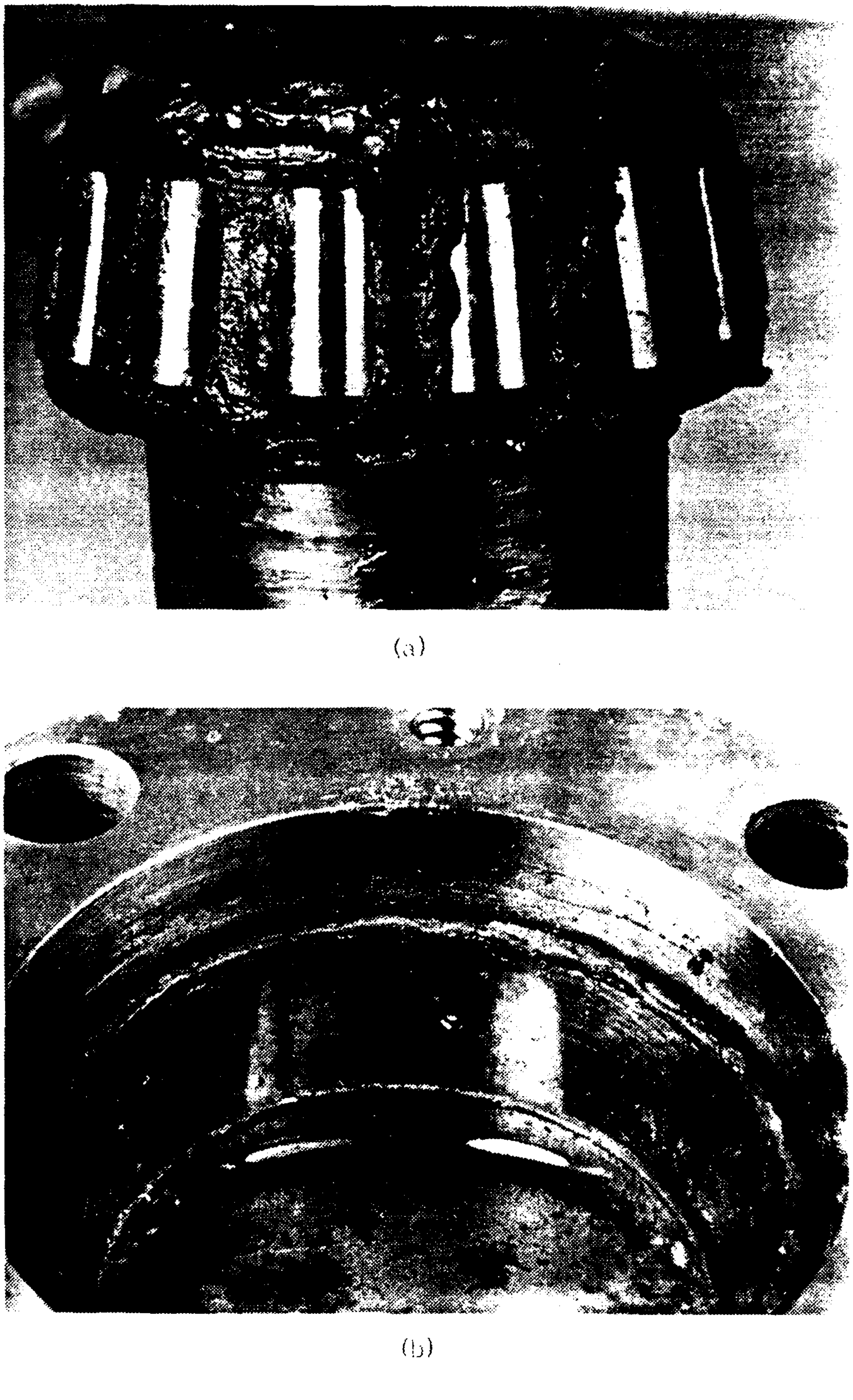


(a)



(b)

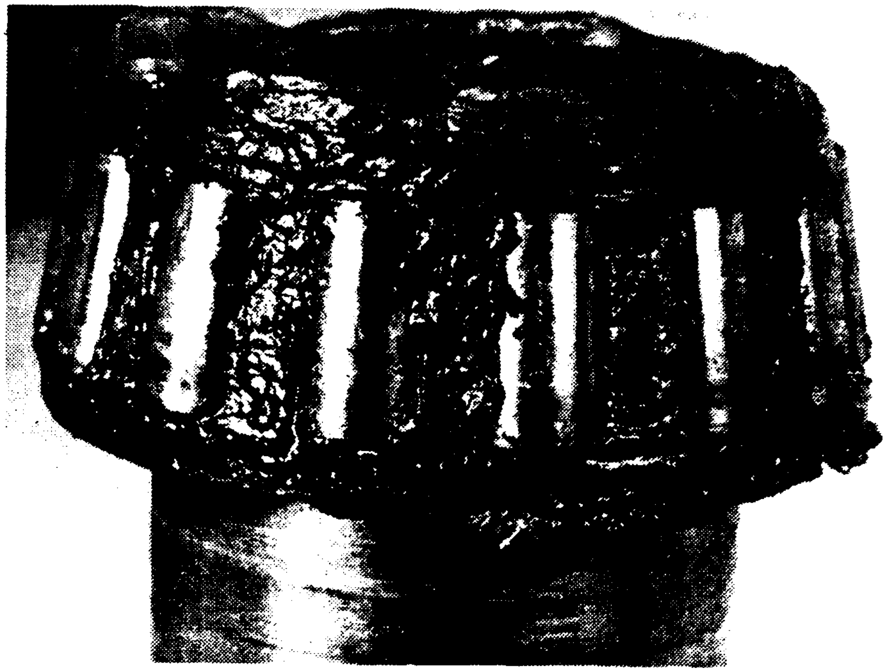
Figure [illegible]. Appearance of same bearing as in Figure 12 (test grease M-720D) after 2 [illegible] with no seawater added.



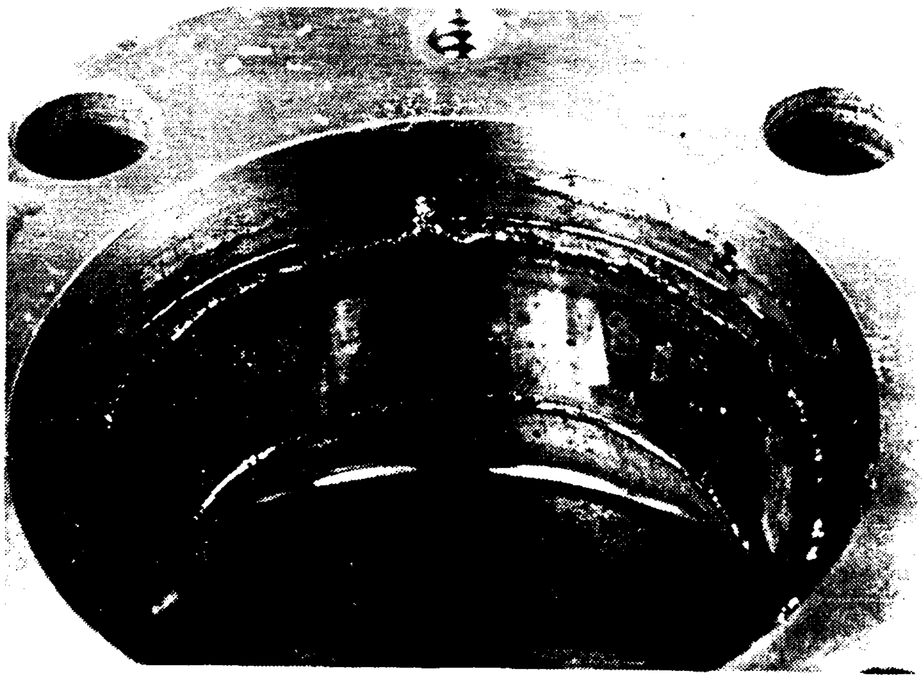
(a)

(b)

Figure 14. Appearance of same bearing [illegible] in Figure 12 (test grease M-[illegible]) after 2 hr in [illegible] and 2 hr in seawater. Brown rust [illegible] formed.



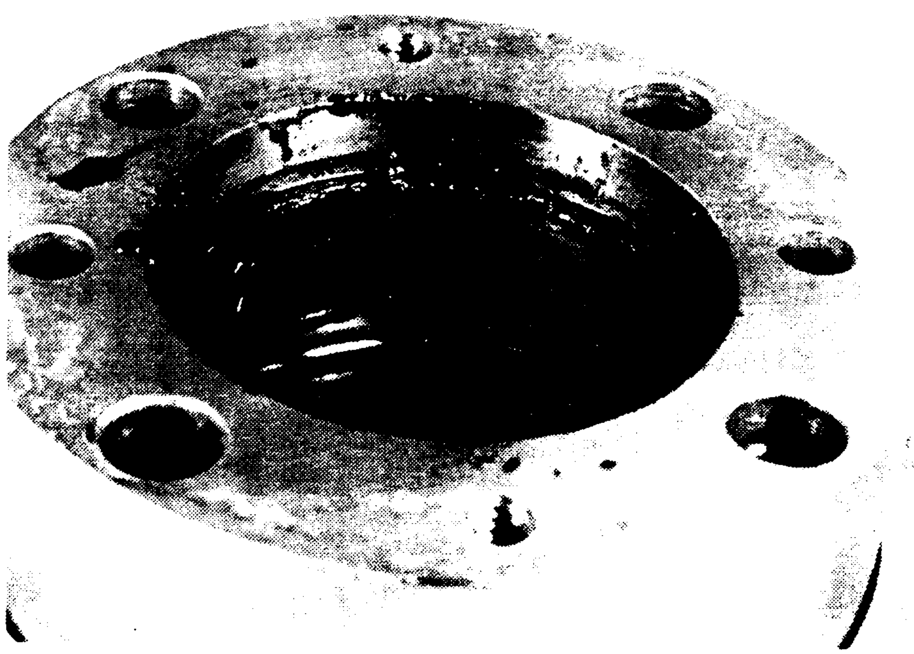
(a)



(b)

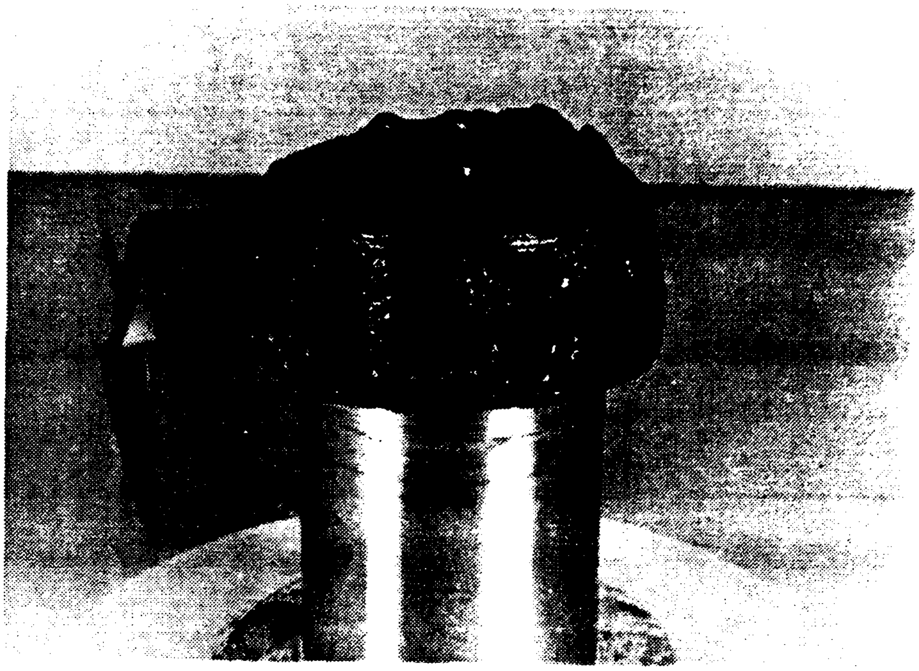
Figure 15. Appearance of same bearings as in Figure 12 (test grease M-7791) after 2 h run-in and 4 h in seawater. Grease contains mostly brown and some black rust.

(a)

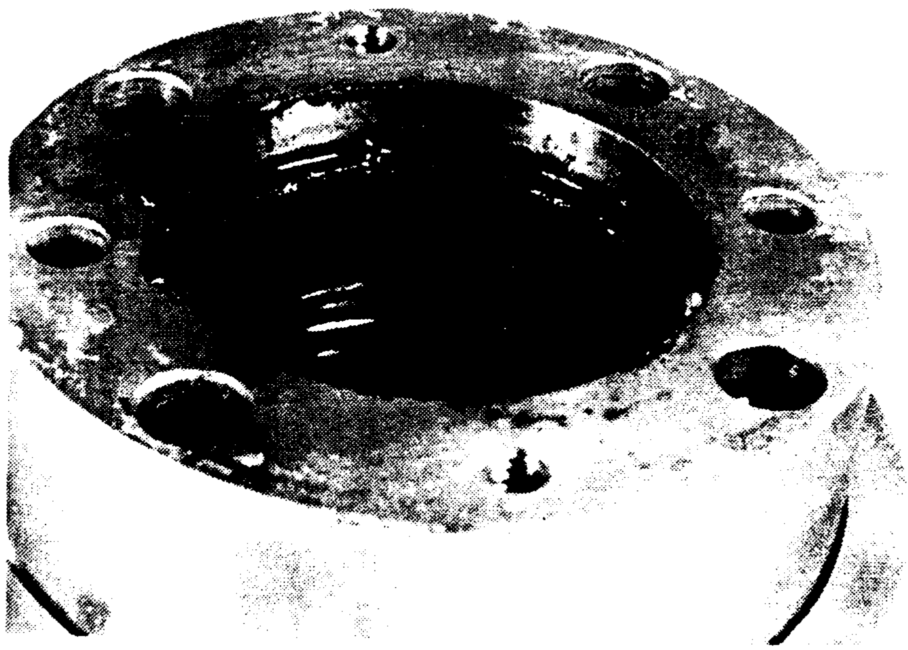


(b)

Figure 16. Appearance of same bearings as in Figure 12 (test grease M-7701) after 2 h run-in and 6 h in seawater. Grease has turned black, for the most part.

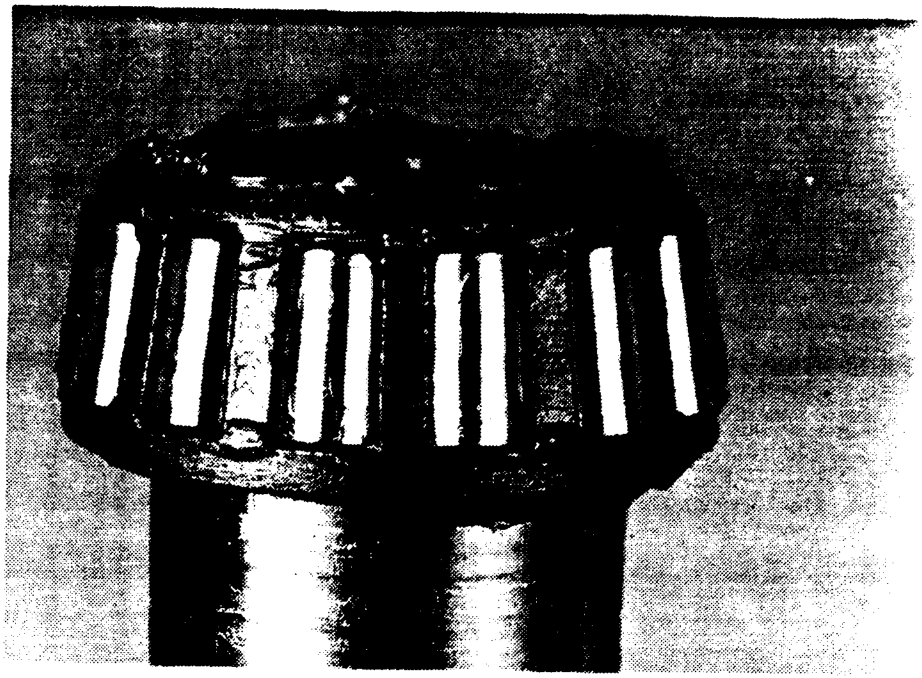


(a)

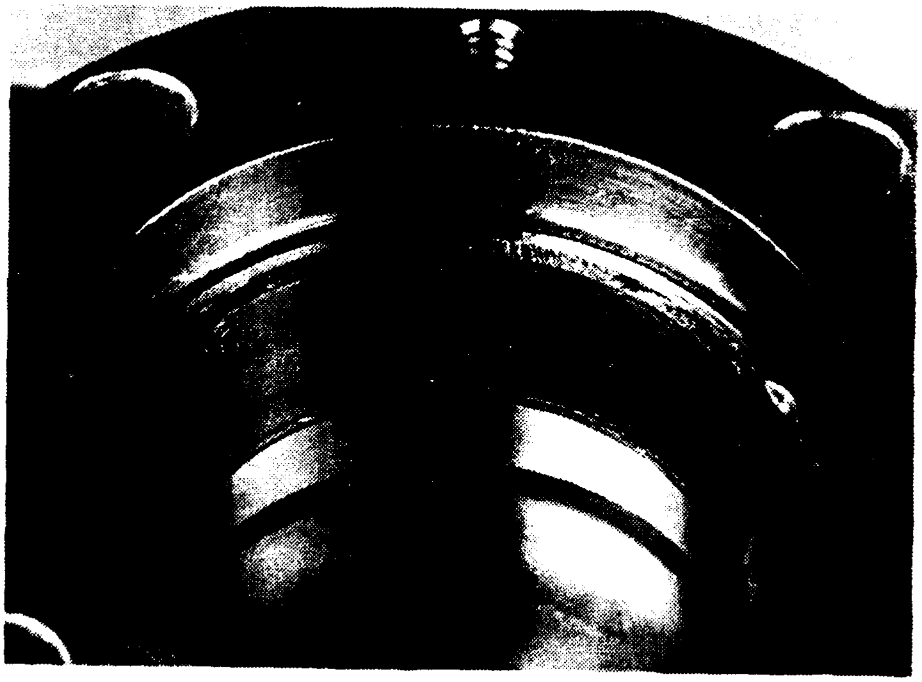


(b)

Figure 17. Appearance of same bearings as in Figure 12 (test grease M-7701) after 2 h run-in and 9 h in seawater. Grease has turned totally black.

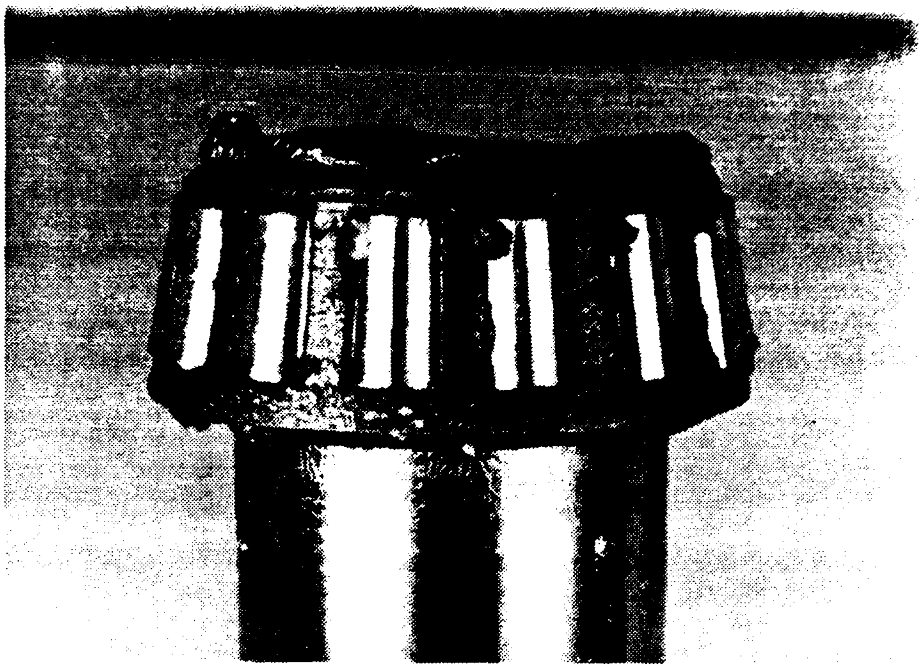


(a)

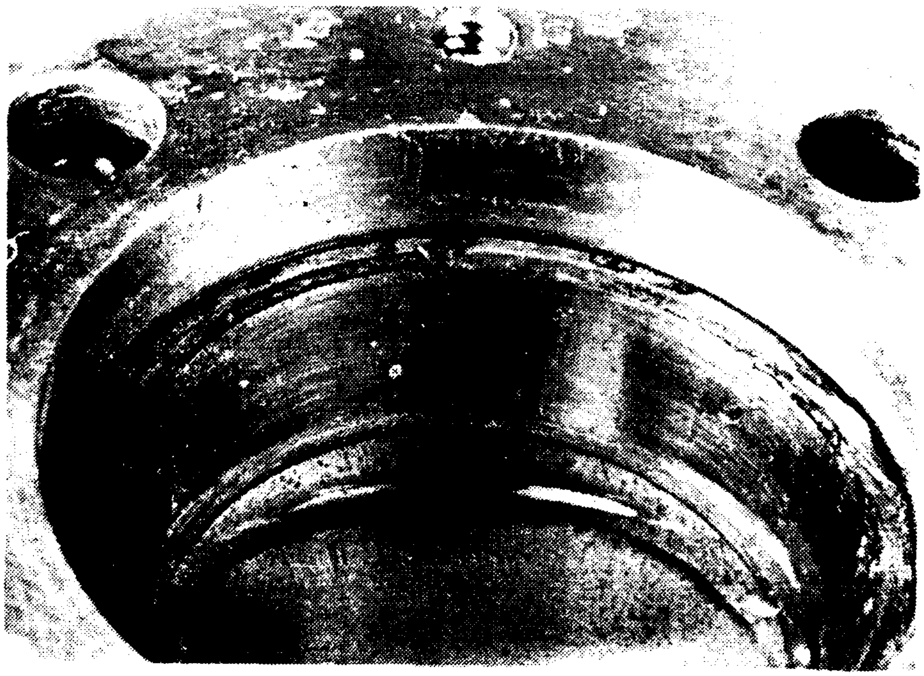


(b)

Figure 18. Appearance of test bearings (cup and greased cone) with test grease M-7703 after 2 h run-in.

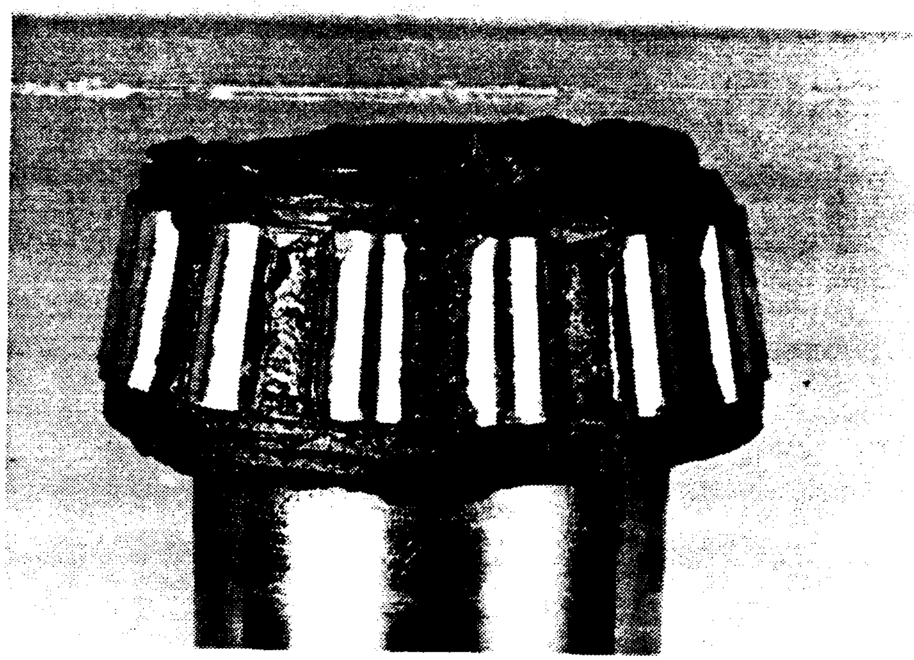


(a)

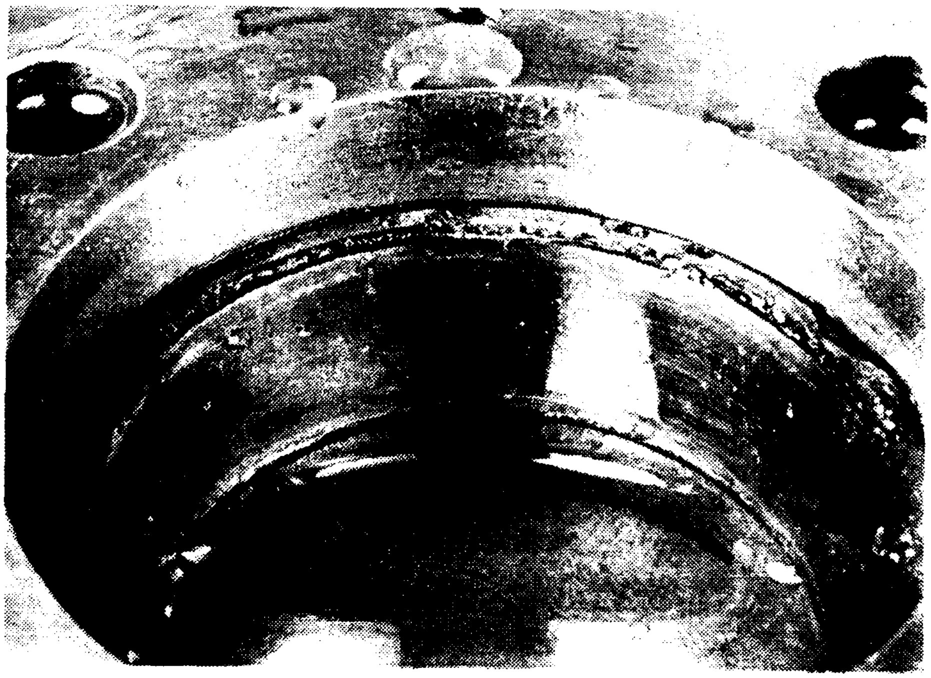


(b)

Figure 19. Appearance of same bearings as in Figure 18 (test grease M-7703) after 2 h run-in and 2 h in seawater. Grease color seems unchanged.

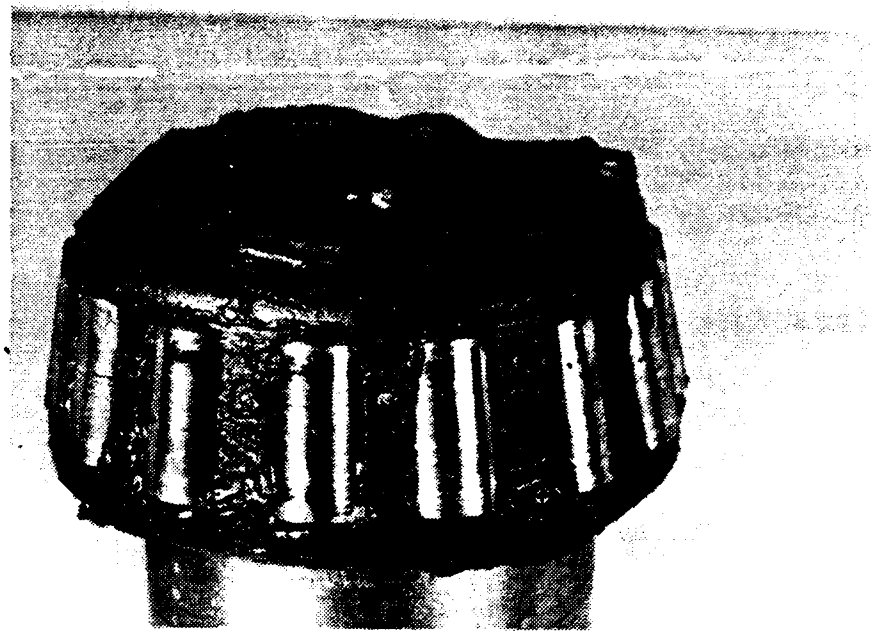


(a)

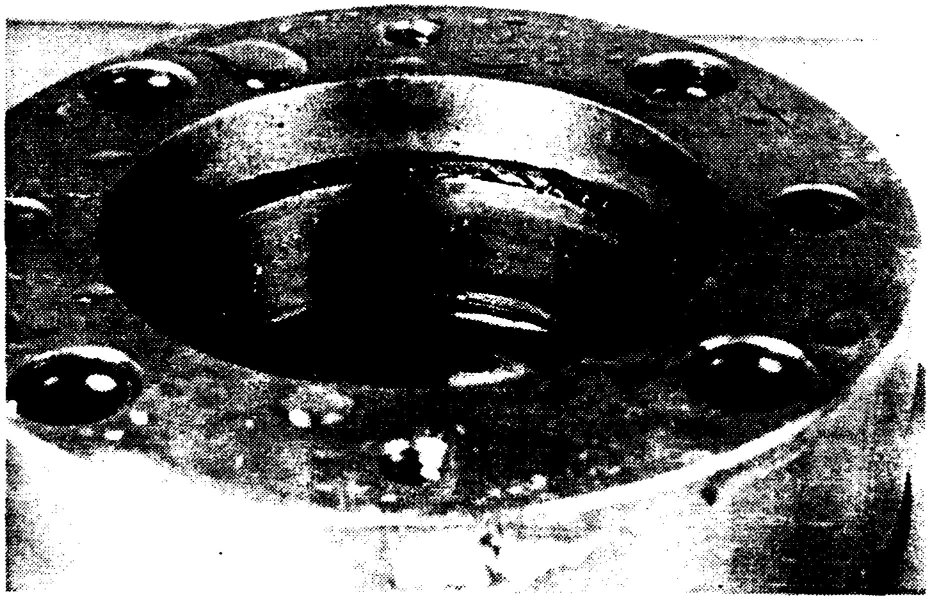


(b)

Figure 20. Appearance of same bearings as in Figure 18 (test grease M-7703) after 2 h run-in and 4 h in seawater. Very small increase in brown rust, also slight change in color of grease.

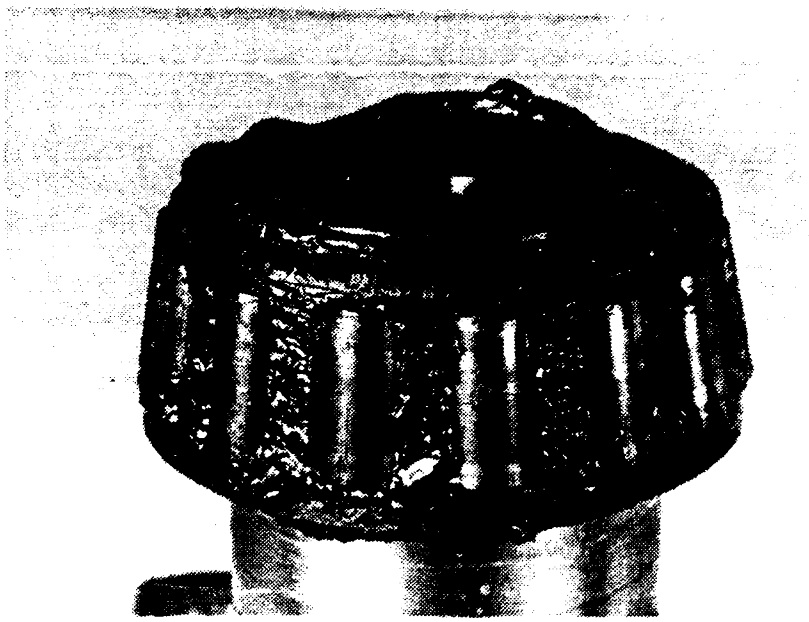


(a)

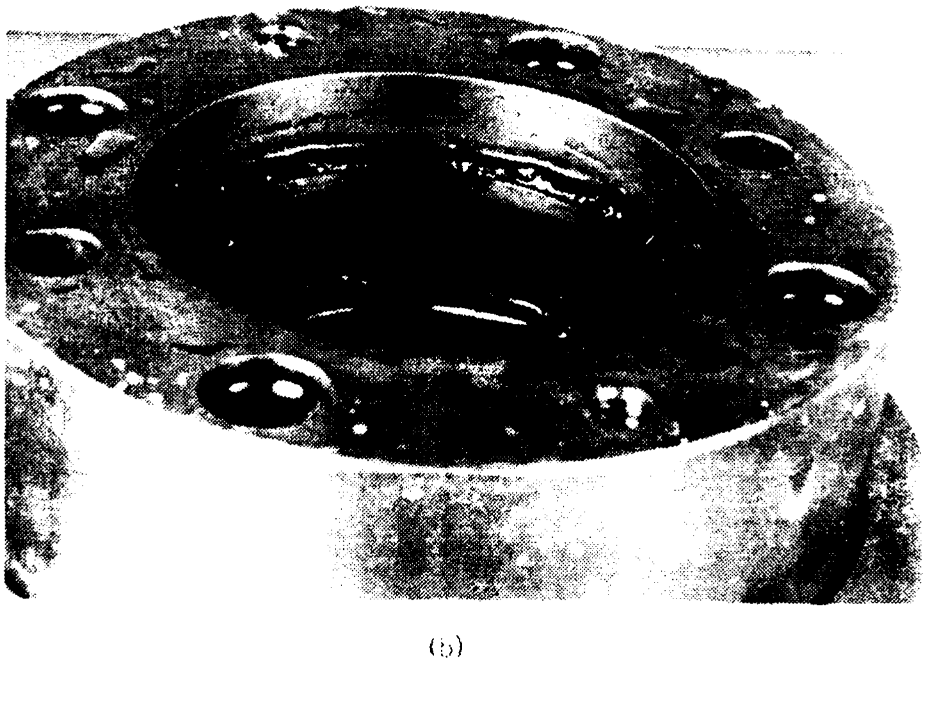


(b)

Figure 21. Appearance of same bearings as in Figure 13 (test grease N° [illegible] after 2 h run-in and 7 h in seawater. Color of grease changed to brown.

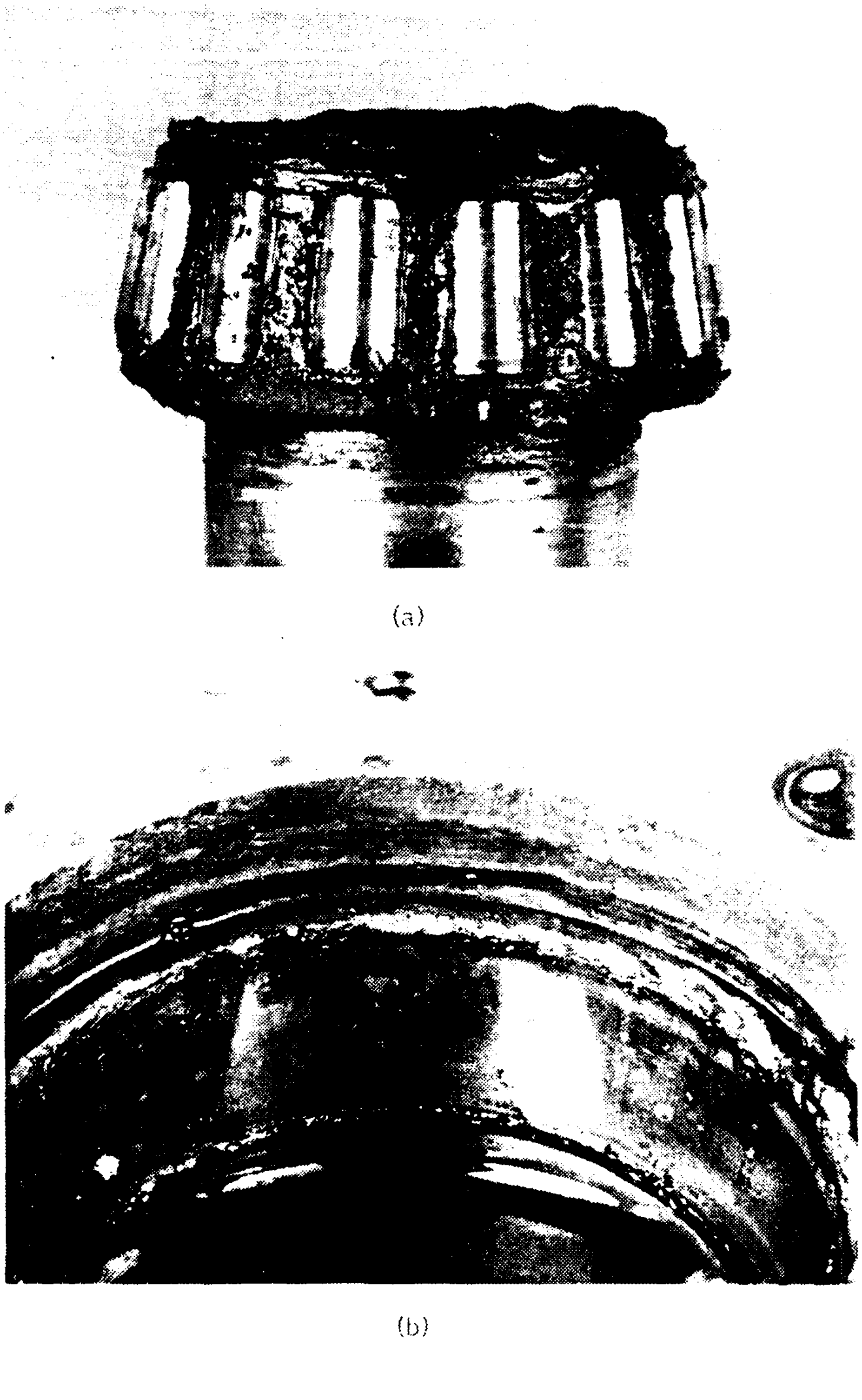


(a)



(b)

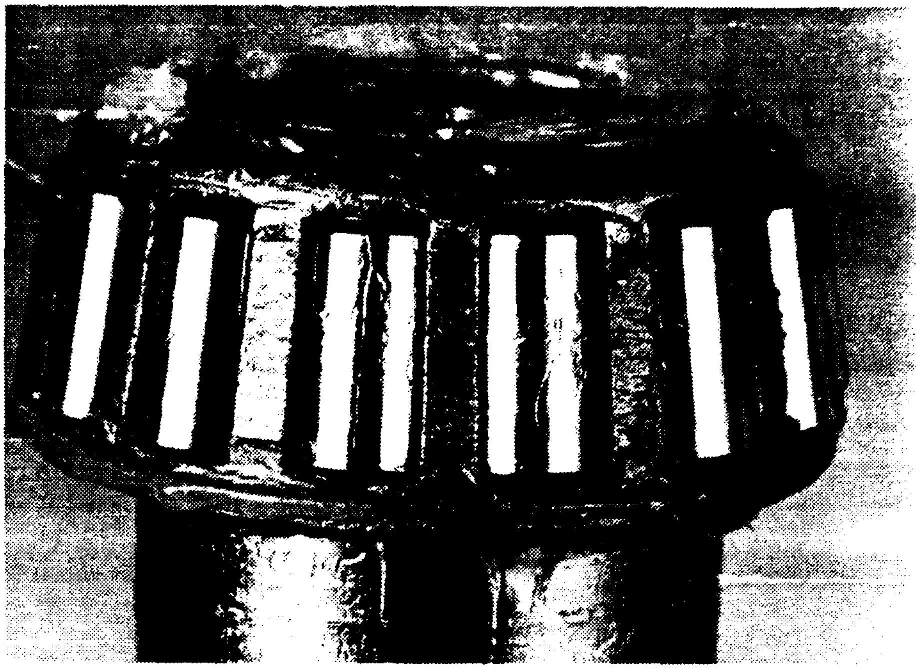
Figure 22. Appearance of same bearings as in Figure 18 (test grease M-7703) after 2 h run-in and 10 h in seawater. Small amount of black rust has formed.



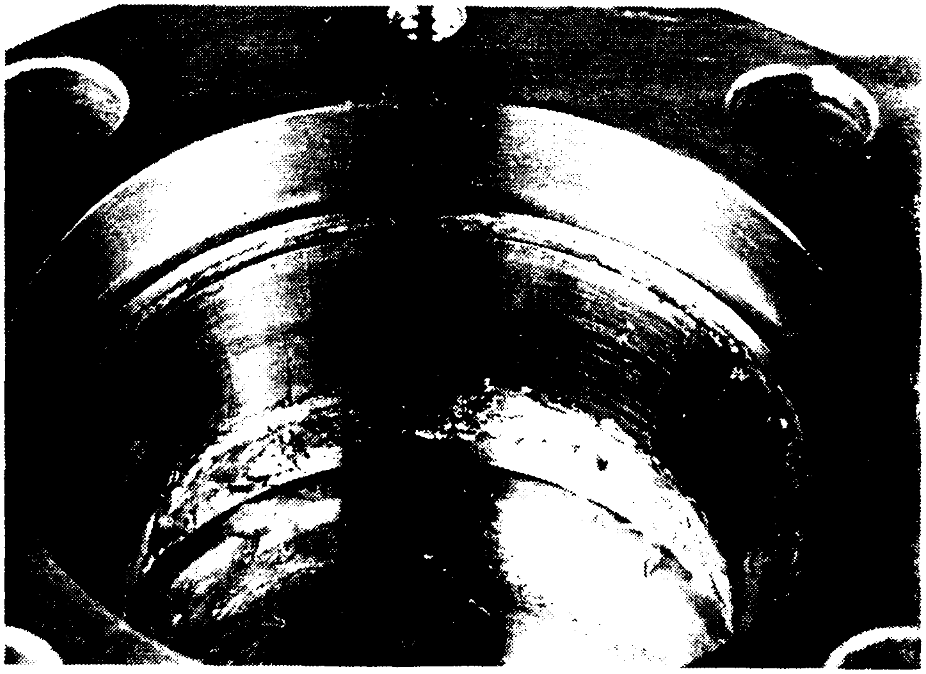
(a)

(b)

Figure 23. Appearance of same bearings as in Figure 18 (test grease M-7703) after 2 h run-in and 26 h in seawater. Grease appears to have turned black.



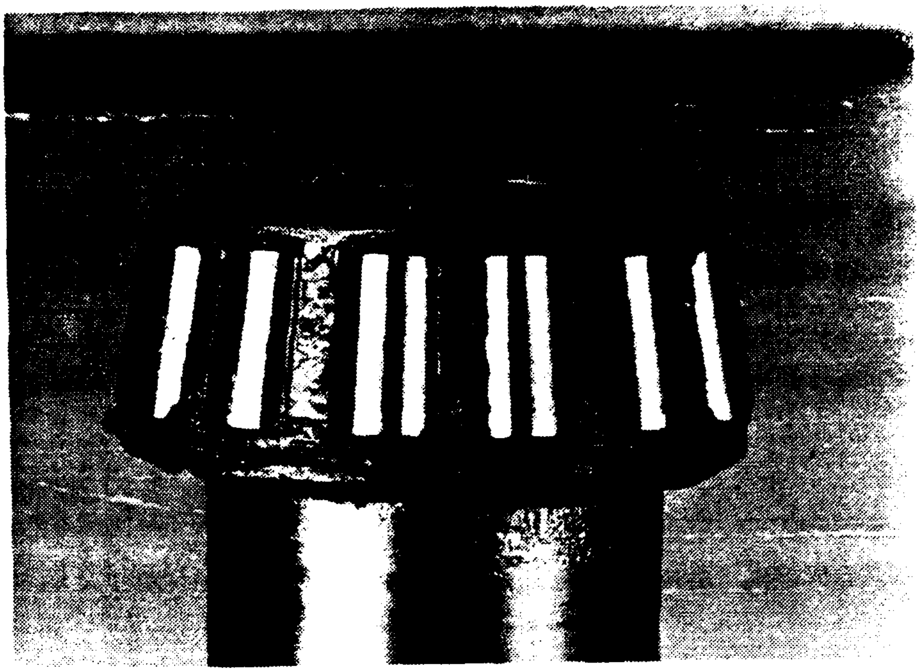
(a)



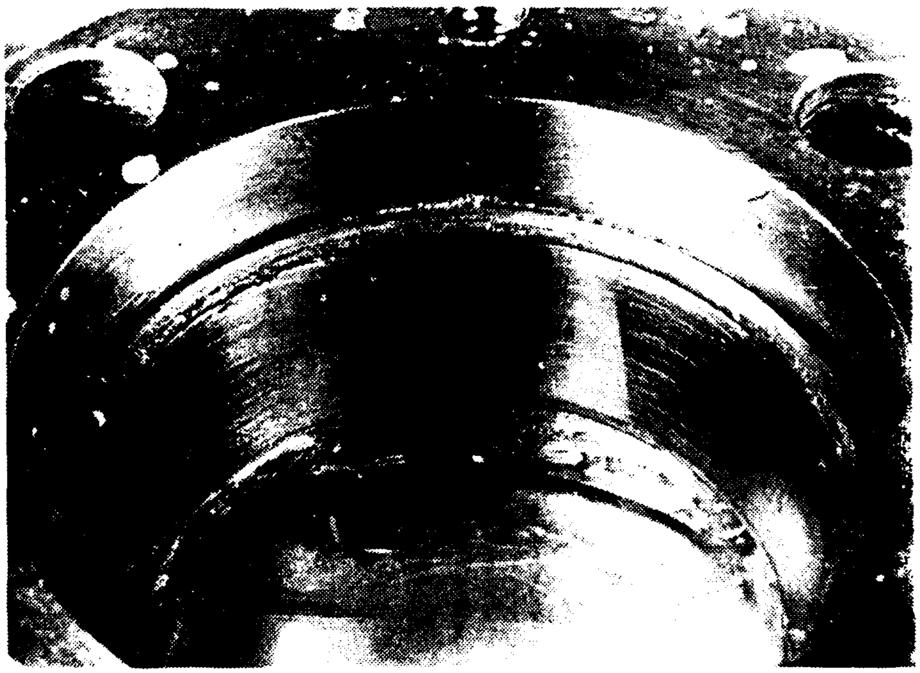
(b)

Figure 2[illegible]. [illegible] (cup and greased cone) with test grease M-7707 after 2 h run-in.

[illegible]

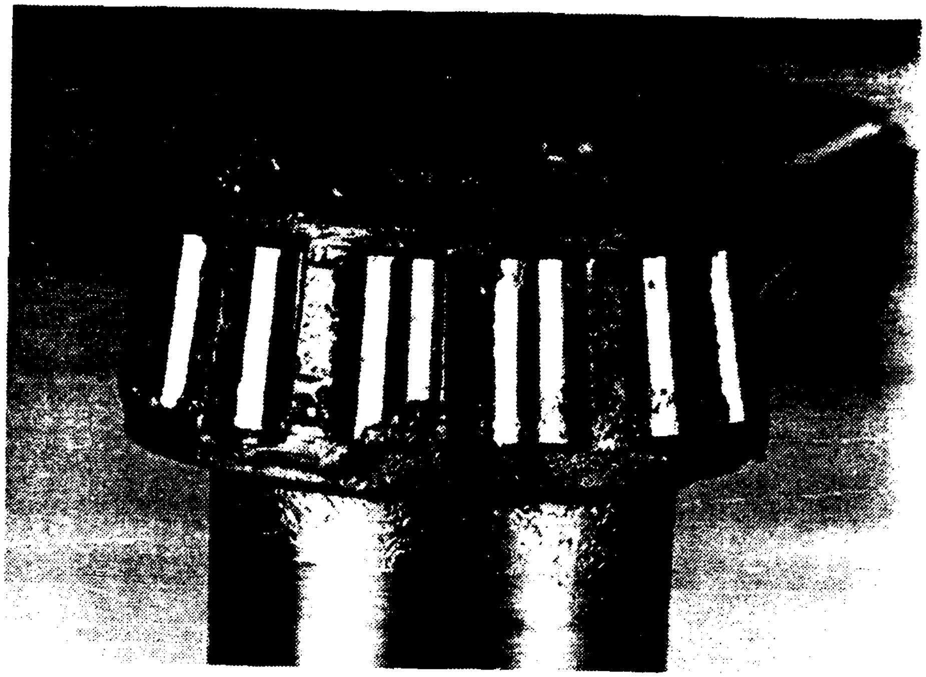


(a)

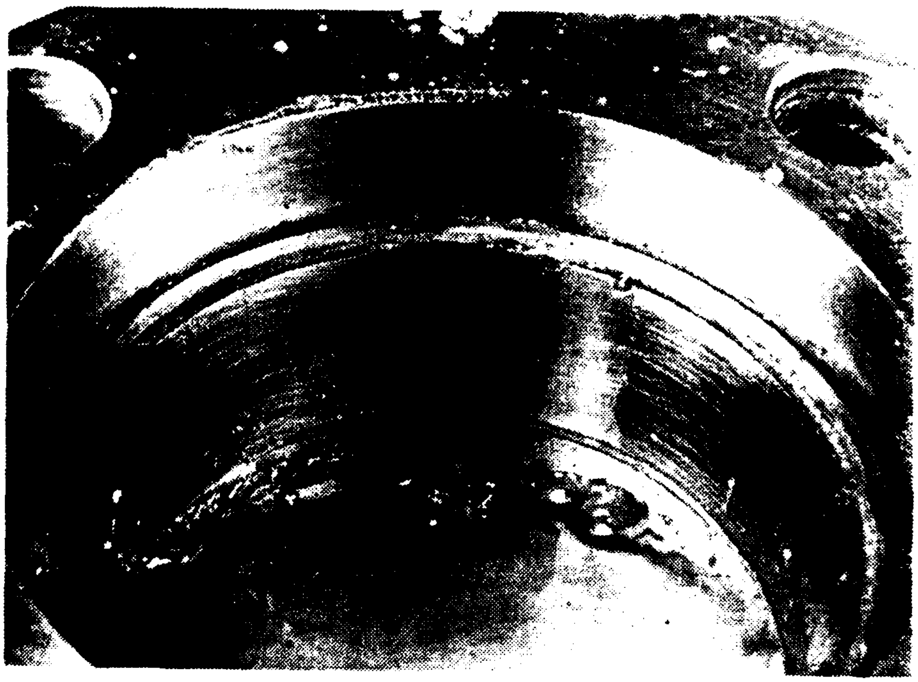


(b)

Figure 25. Appearance of same bearings as in Figure 24 (test grease M-7707) after 2 h run-in and 2 [illegible] in seawater. No rust formation evident.

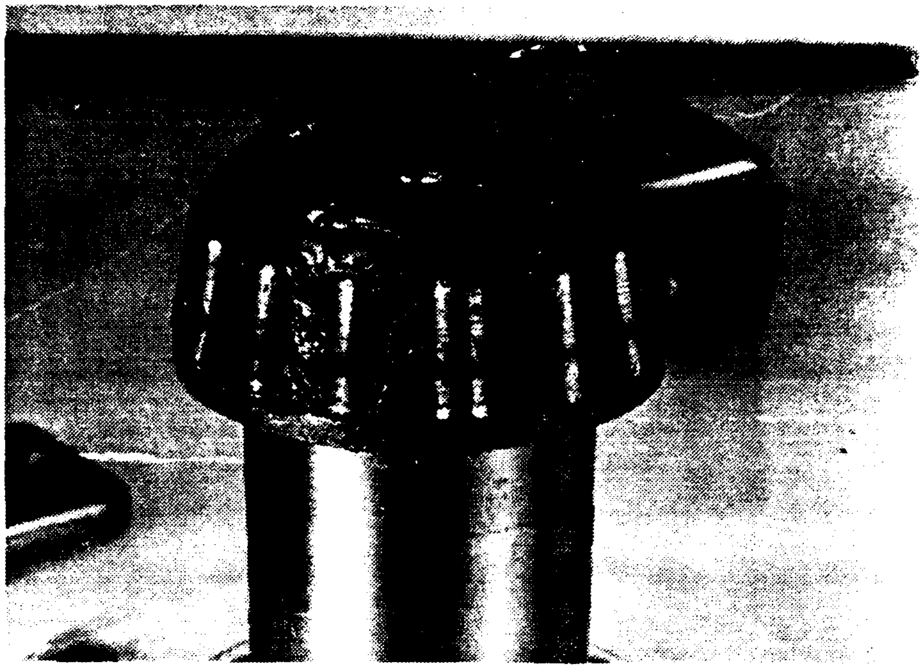


(a)

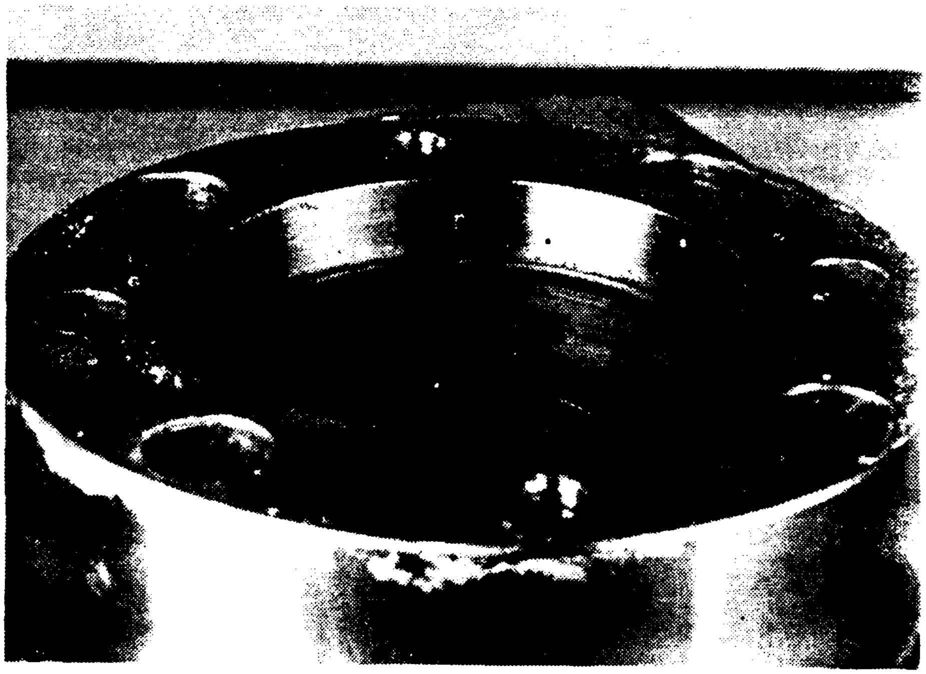


(b)

Figure 26. Appearance of same bearings as in Figure 24 (test grease M-7707) after 2 h run-in and 4 h in seawater. Very little rust formation is visible.

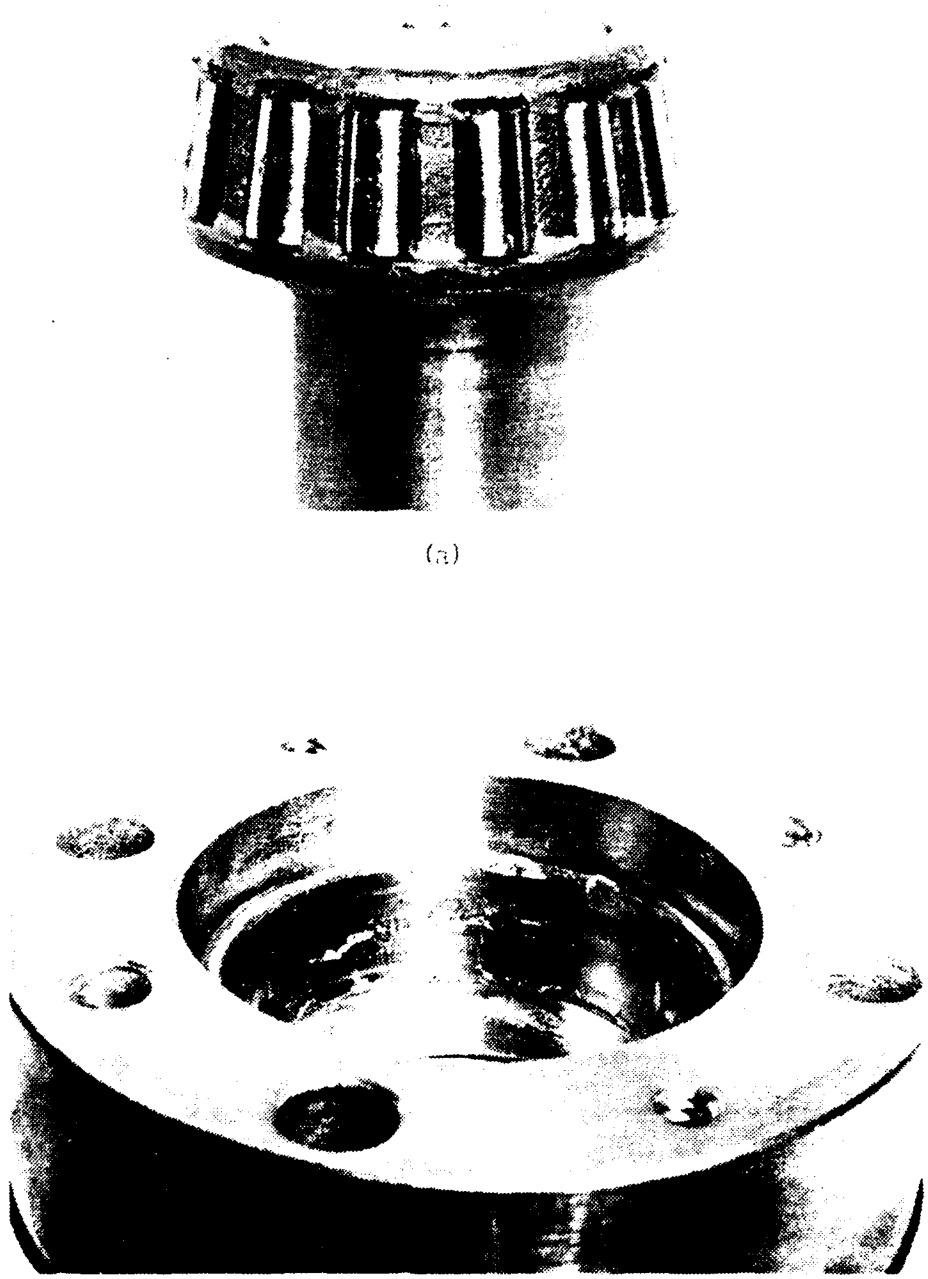


(a)



(b)

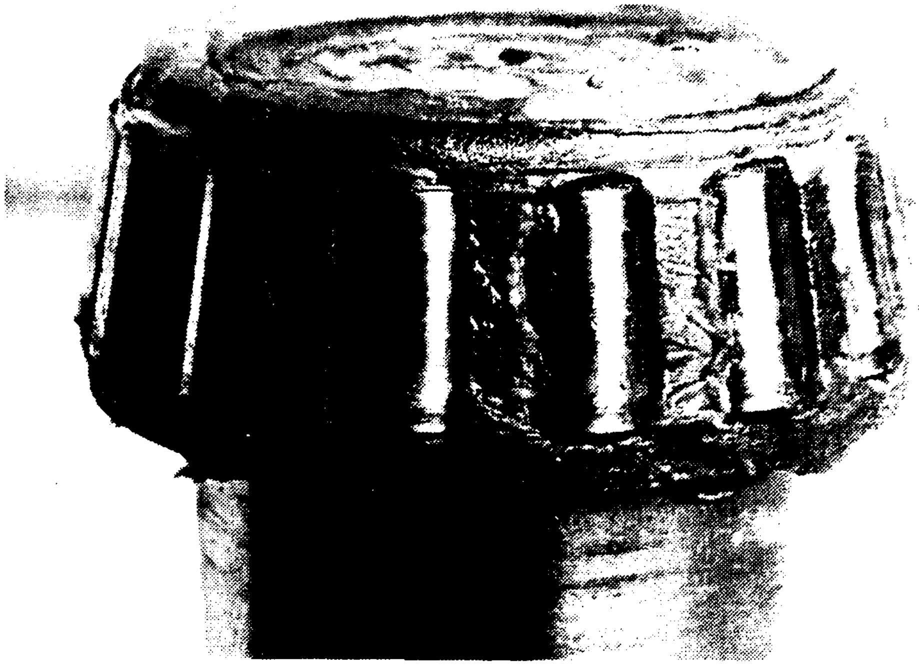
Figure 27. Appearance of same bearings as in Figure 24 (test grease M-7767) after 2 h run in and 7 h in seawater. Substantial amount of brown and black rust has formed.



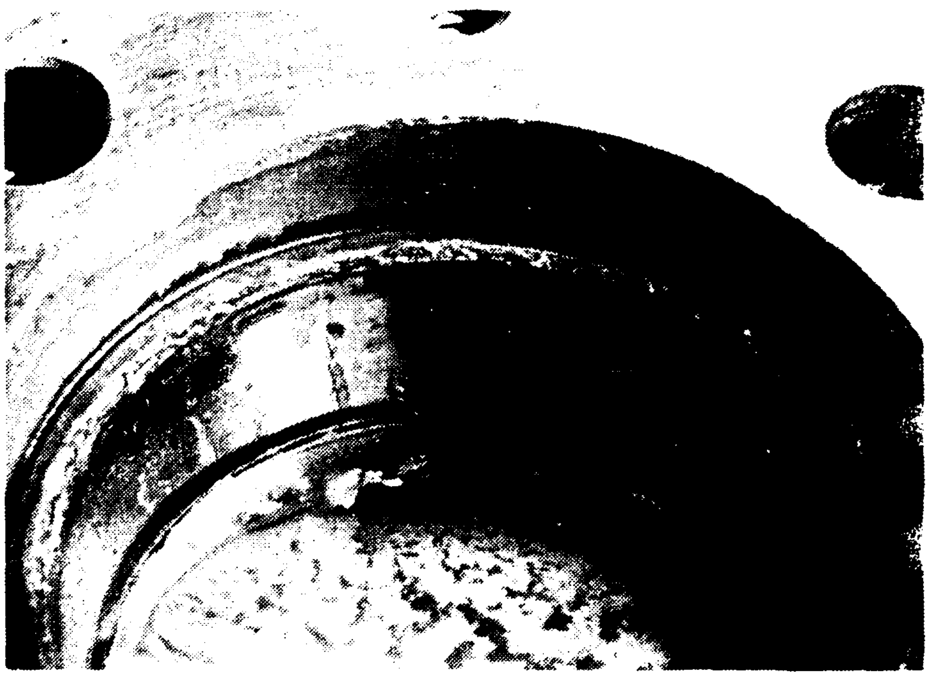
(a)

(b)

Figure 28. Appearance of test bearings (cup and greased cone) with specially formulated grease M-7707 ND (without a binder) after 2 h run-in.

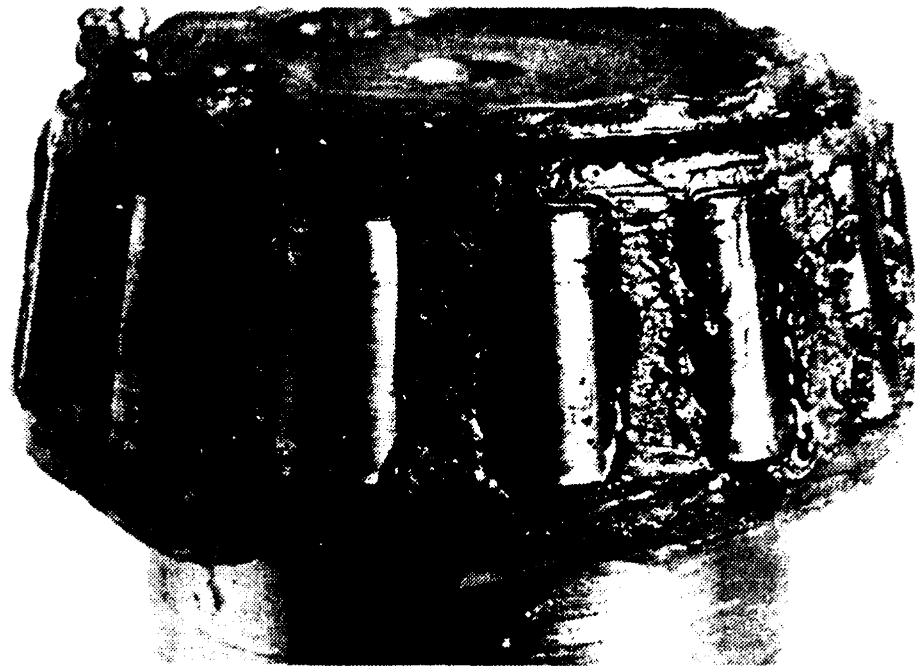


(a)



(b)

Figure 29. Appearance of same bearings as in Figure 28 (test grease M-7707-ND) after 2 h run in and 2 h in seawater. Substantial amount of brown rust formed.



(a)

(b)

Figure 33. Appearance of same bearings as in Figure 28 (test grease M-7767-ND) after 2 h run-in and 4 h in seawater. Formation of black rust is shown.

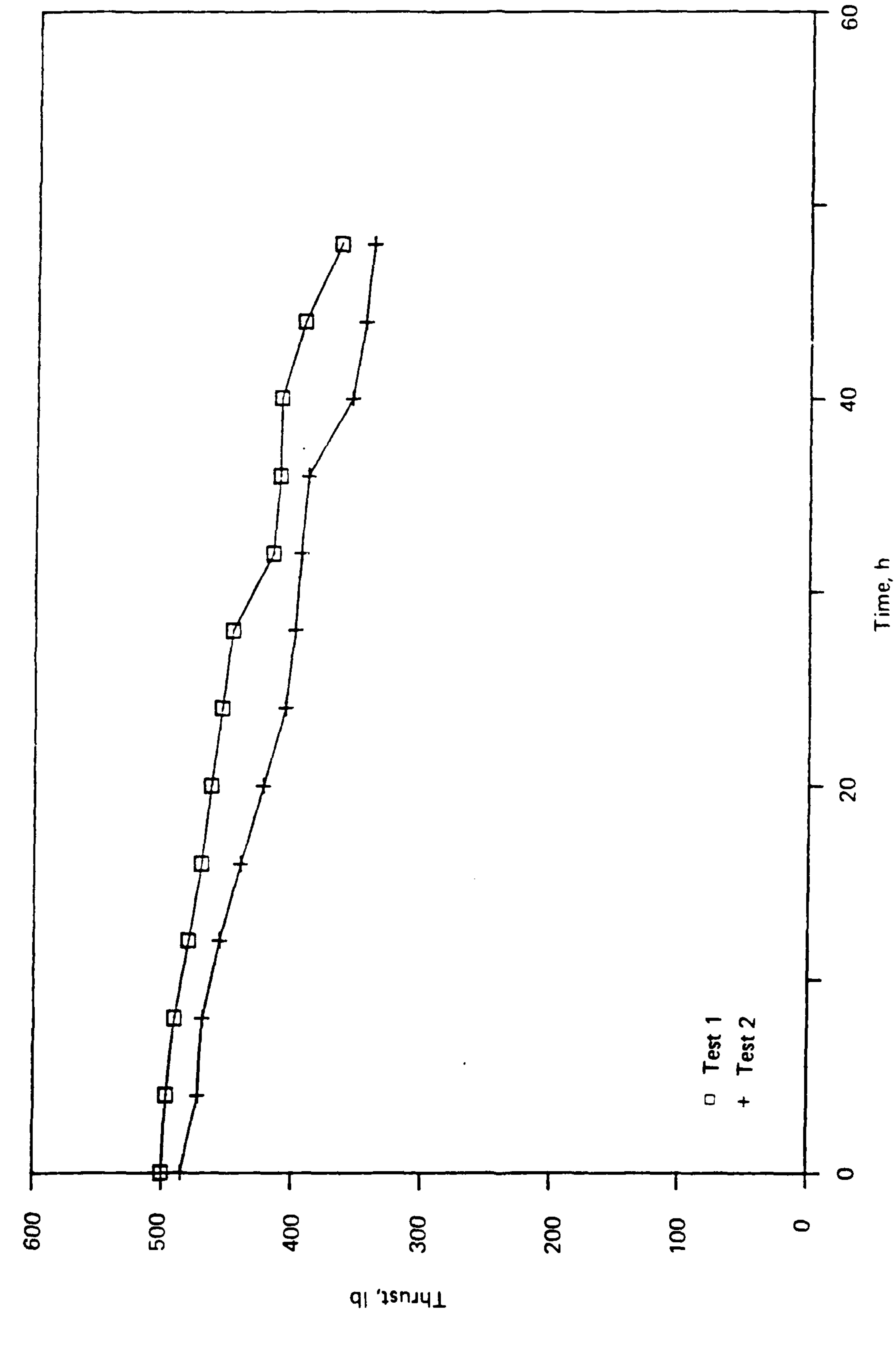


Figure 31. Thrust drop curves for grease M-7701.

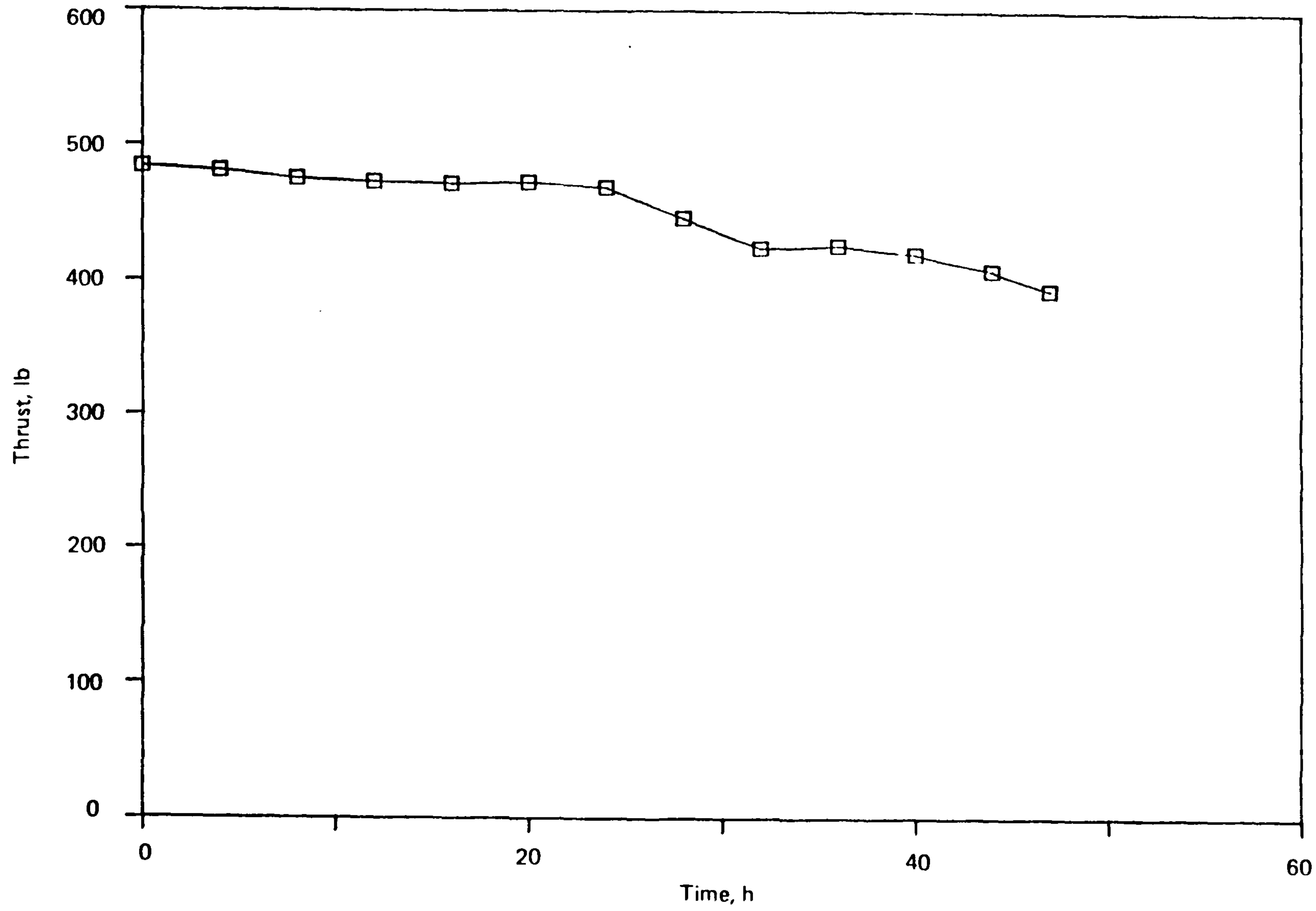


Figure 32. Thrust drop curve for grease M-7703.

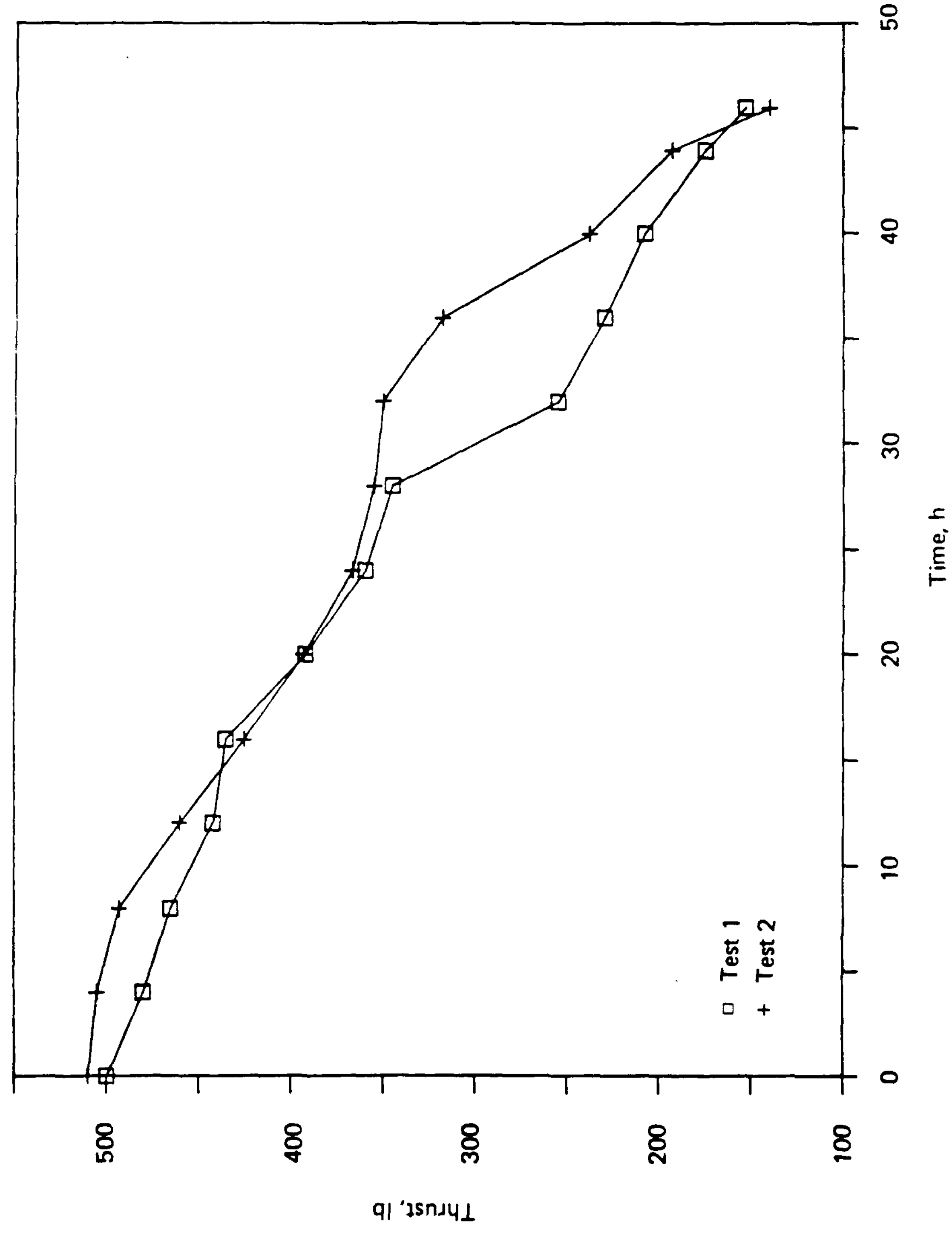


Figure 33. Thrust drop curves for specially formulated grease M-7707-NI containing no inhibitor.

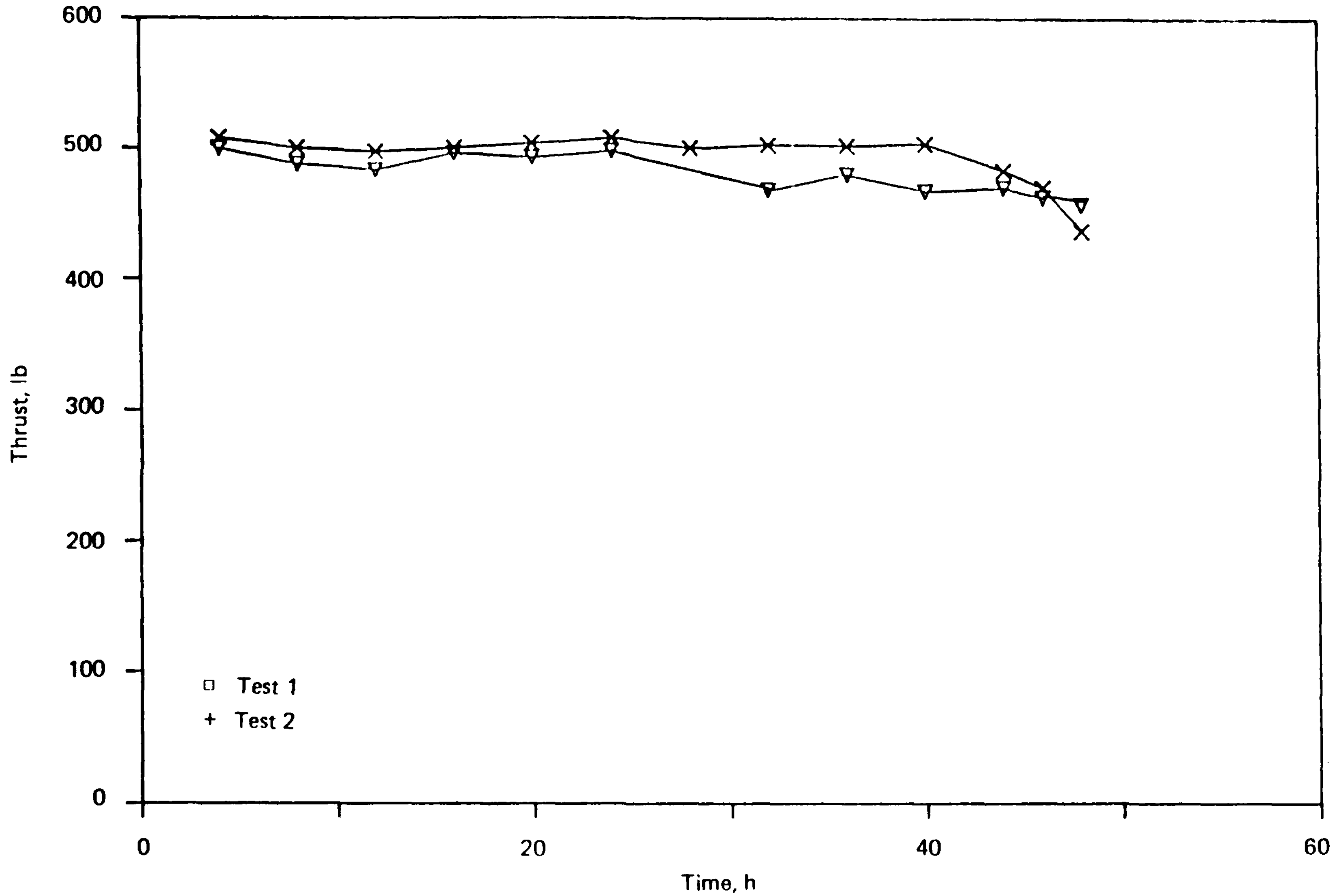


Figure 34. Thrust drop curves for the experimental Chevron SRI grease 2.

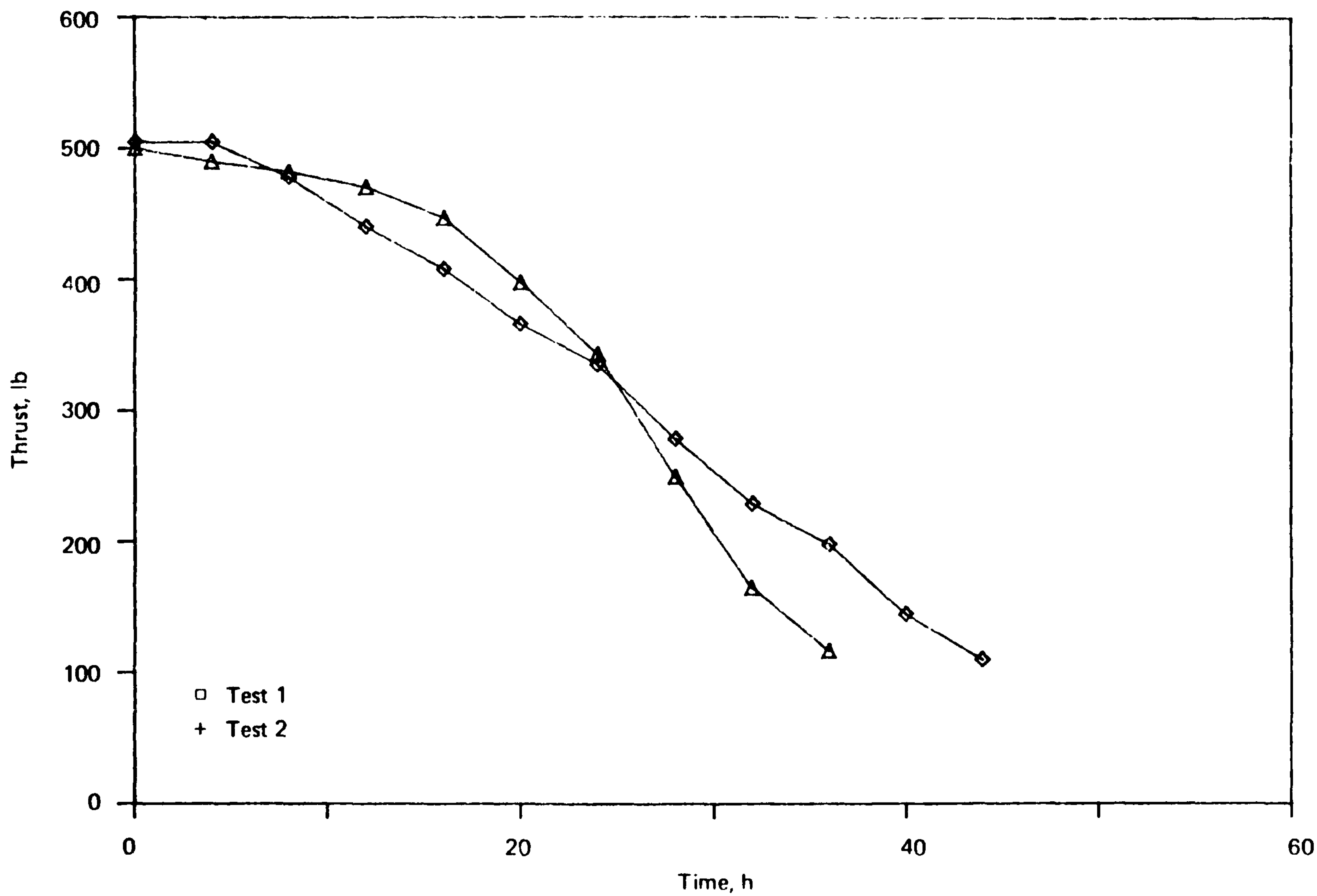


Figure 35. Thrust drop curves for the experimental grease Mobil WTR (81322).

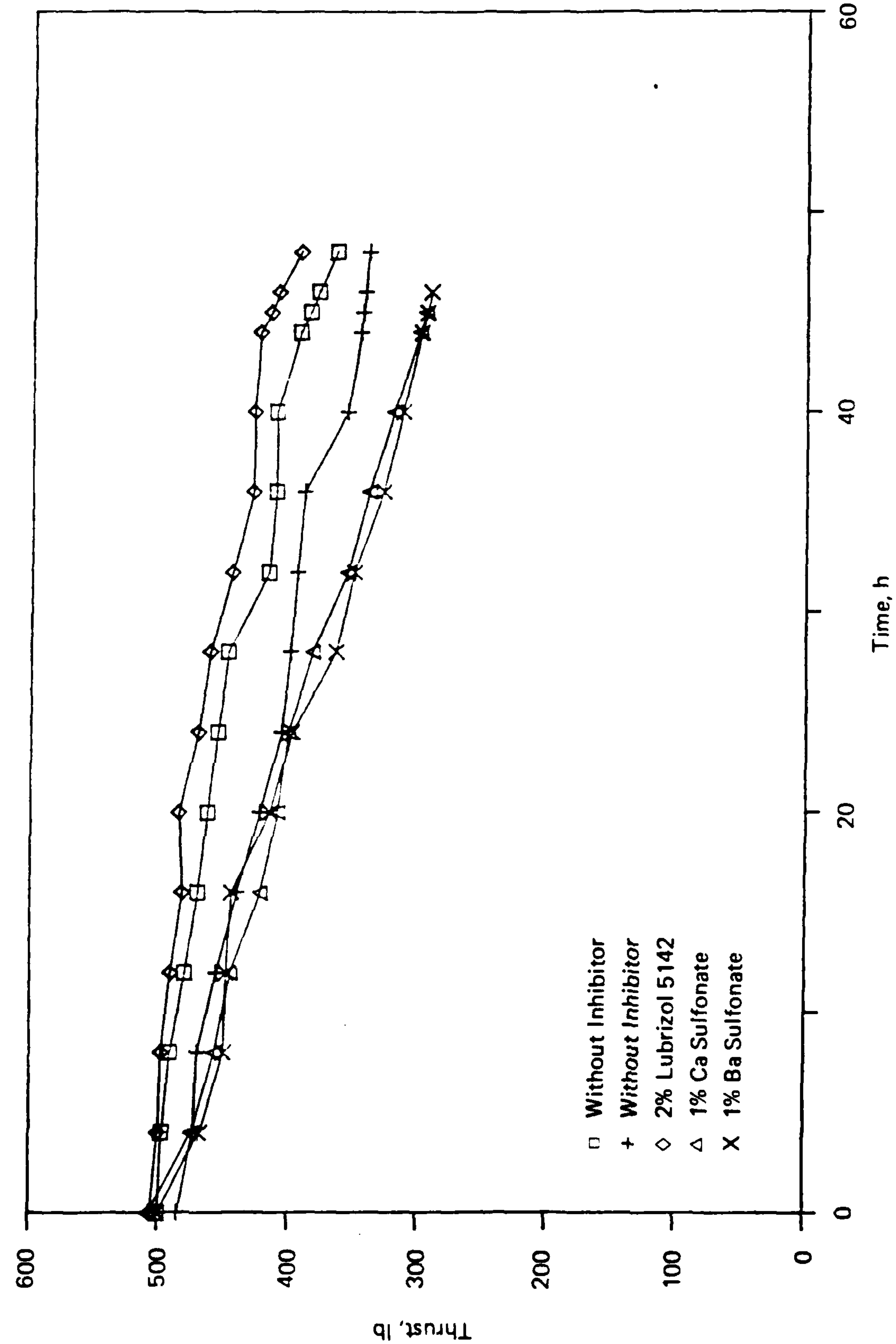


Figure 36. Thrust drop curves for grease M-7701 with and without added inhibitors.

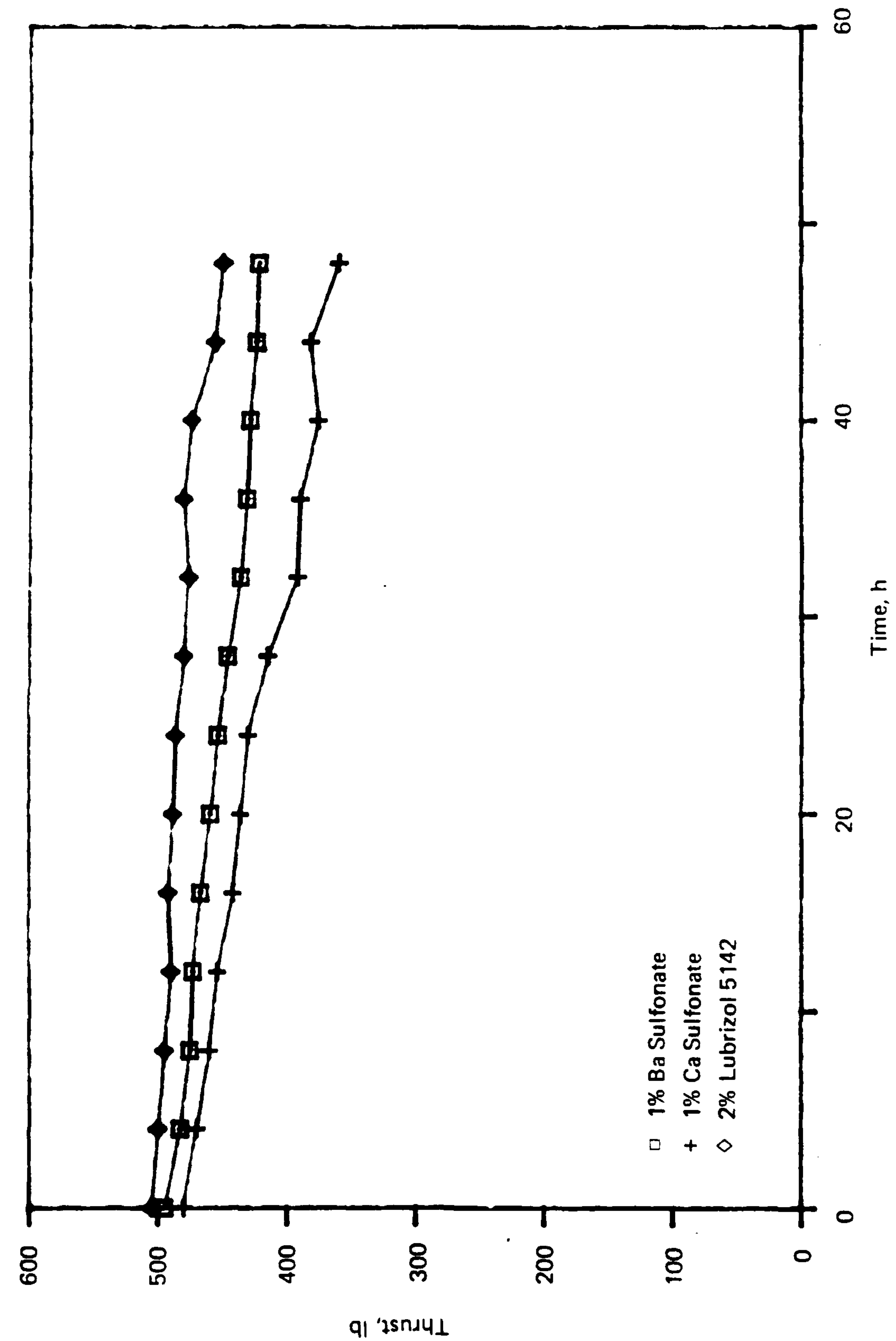


Figure 37. Thrust drop curves for grease M-7707 with added inhibitor.

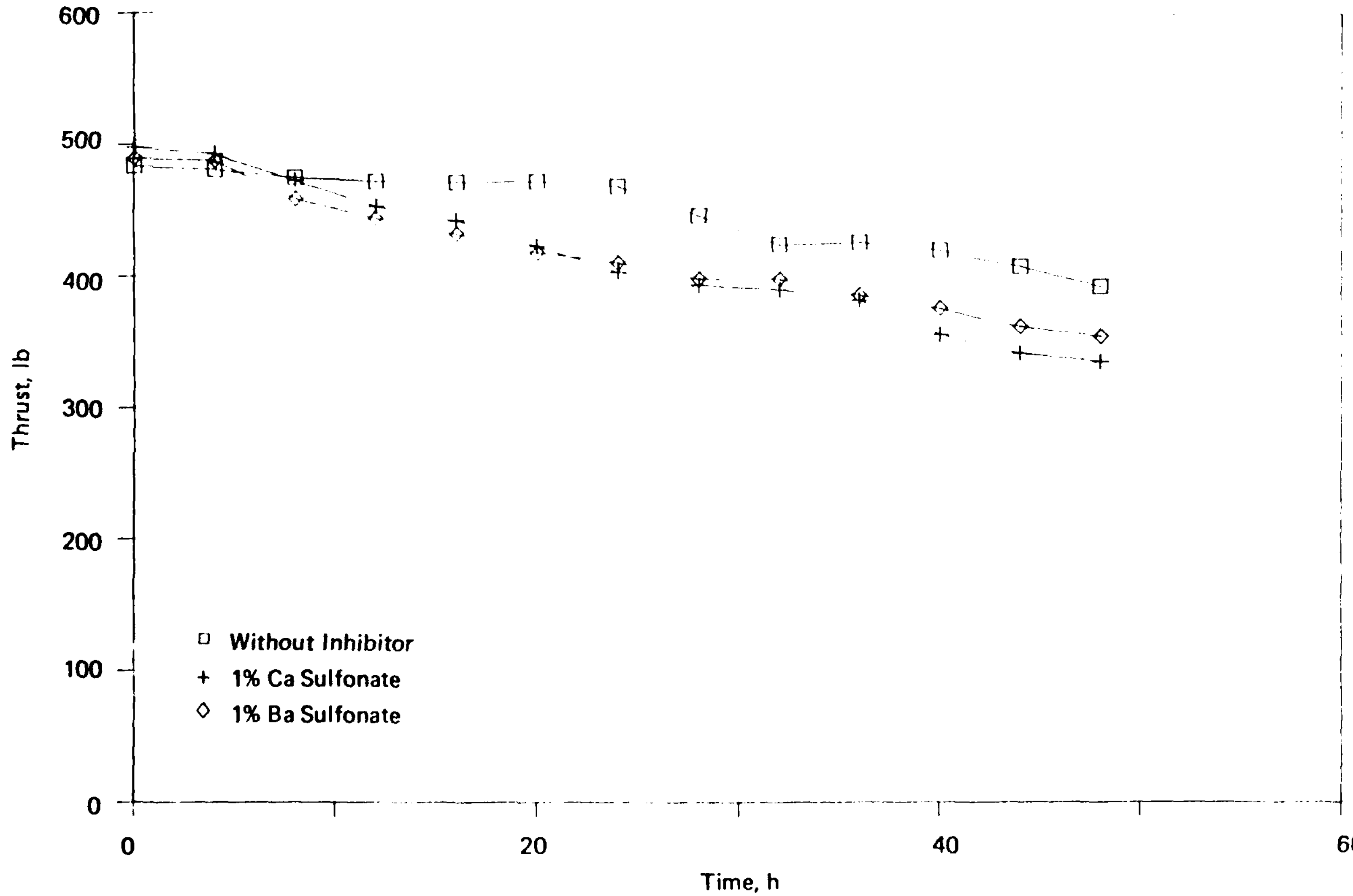


Figure 38. Thrust drop curves for grease M-7703 with and without added inhibitors.